



NASA TN D-8046

N76-13268

Unclas

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Cleveland, Ohio 44135



1. Report No. NASA TN D-8046	2. Government Accession No.	3. Recipient's Catalog No.	
4. Title and Subtitle HYDROGEN ENVIRONMENT EMBRITTLEMENT OF ASTROLOY AND UDIMET 700 (NICKEL-BASE) AND V-57 (IRON-BASE) SUPERALLOYS		5. Report Date December 1975	
		6. Performing Organization Code	
7. Author(s) Hugh R. Gray and Joseph P. Joyce		8. Performing Organization Report No. E-8407	
9. Performing Organization Name and Address Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio 44135		10. Work Unit No. 506-23	
		11. Contract or Grant No.	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546		13. Type of Report and Period Covered Technical Note	
		14. Sponsoring Agency Code	
15. Supplementary Notes			
16. Abstract <p>The sensitivity to hydrogen environment embrittlement of three superalloys was determined. Astroloy forgings were resistant to embrittlement during smooth tensile, notched tensile, and creep testing in 3.5-MN/m² hydrogen over the range 23° to 760° C. The notched tensile strength of Udimet 700 bar stock in hydrogen at 23° C was only 50 percent of the baseline value in helium. Forgings of V-57 were not significantly embrittled by hydrogen during smooth tensile testing over the range 23° to 675° C. However, creep and rupture lives of V-57 were degraded by hydrogen. Postcreep tensile ductility of V-57 was reduced by 40 percent after creep exposure in hydrogen.</p>			
17. Key Words (Suggested by Author(s)) Hydrogen; Hydrogen embrittlement; Hydrogen environment embrittlement; Superalloys; Udimet 700; V-57; Hydrogen embrittlement of superalloys		18. Distribution Statement Unclassified - unlimited STAR category 26 (rev.)	
19. Security Classif (of this report) Unclassified	20. Security Classif (of this page) Unclassified	21. No. of Pages 45	22. Price* \$3.75

HYDROGEN ENVIRONMENT EMBRITTLEMENT OF ASTROLOY AND UDIMET 700 (NICKEL-BASE) AND V-57 (IRON-BASE) SUPERALLOYS*

by Hugh R. Gray and Joseph P. Joyce

Lewis Research Center

SUMMARY

One purpose of this investigation was to resolve the differences in the literature regarding the degree of susceptibility to hydrogen environment embrittlement of the two compositionally similar alloys Udimet 700 and Astroloy. Another purpose was to evaluate superalloys that are candidates for use as turbine disks in hydrogen-oxygen auxiliary power units for resistance to embrittlement by hydrogen.

Udimet 700 and Astroloy (nickel-base superalloys) and V-57 (an austenitic iron-base superalloy) were mechanically tested in hydrogen at pressures of 3.5 and 1.7 MN/m², respectively. Notched and smooth tensile tests were conducted over the temperature range 23° to 760° C. Creep tests were conducted at 565° and 675° C and were followed by postcreep tensile tests at 23° C to determine residual strength and ductility.

Astroloy forgings were relatively immune to hydrogen environment embrittlement. Both smooth and notched tensile strengths of Astroloy in hydrogen were at least 92 percent of air values over the range 23° to 760° C. Limited creep testing of Astroloy did not indicate any degradation due to hydrogen. The fine grain size and fine γ' precipitate size resulting from forging and heat treatment were presumably responsible for the good performance exhibited by Astroloy in hydrogen.

Udimet 700 bar stock exhibited reductions of approximately 50 percent in notched tensile strength at 23° C when tested in hydrogen. This sensitivity of Udimet 700 to hydrogen environment embrittlement was presumably related to its large grain size and large γ' precipitate size resulting from conventional heat treatments.

Forgings of V-57 were not significantly embrittled by hydrogen during tensile testing over the range 23° to 675° C. However, in creep and rupture at 565° and 675° C, the Larson-Miller parameters were about 1 to 2 less in hydrogen than in helium. Post-creep tensile testing at 23° C indicated that residual ductility of V-57 was decreased approximately 40 percent by creep testing in hydrogen.

* Presented in part at AIME Conference on Effect of Hydrogen on Behavior of Materials, Moran, Wyoming, September 7-11, 1975.

INTRODUCTION

Initially, hydrogen and oxygen were selected as common propellants for all the Space Shuttle propulsion and power systems, including the auxiliary power unit (APU). The APU will provide hydraulic and electric power during launch-to-orbit and reentry-to-landing shuttle operations. Subsequently, a hydrazine-fueled APU was selected for the initial shuttle flight vehicles. The successful development of a hydrogen-oxygen APU and its use in advanced shuttle vehicles would permit a payload increase of 500 to 700 kilograms. Hence, the NASA Lewis Research Center has supported the hydrogen-oxygen APU program through its recent completion of an extensive integrated system performance test (ref. 1). In this system oxygen and hydrogen are combusted at a ratio of 0.7 to provide a constant turbine inlet temperature of 815°C . Combustion products, namely, hydrogen plus about 10 percent by volume superheated steam, were expanded across a two-stage axial-flow partial-admission turbine. Combustion pressure was varied between 0.4 and 3 MN/m^2 to vary total flow rate and, hence, modulate shaft power output.

Udimet 700, a nickel-base superalloy, was the initial candidate alloy for both turbine disks. However, concern for potential hydrogen environment embrittlement arose when wrought Udimet 700 was reported (ref. 2) to suffer severe losses in both tensile strength and ductility when tested in gaseous hydrogen (fig. 1). For example, the notched tensile strength of Udimet 700 at room temperature in 52- MN/m^2 hydrogen was only 67 percent of its notched tensile strength in air. Limited test data determined in wet hydrogen indicated a similar degree of embrittlement as in dry hydrogen (fig. 1).

Subsequent data reported (ref. 3) for Astroloy, an alloy with essentially the same chemical composition as Udimet 700, indicated that it was quite resistant to embrittlement during testing in gaseous hydrogen. For example, as shown in figure 2, the notched tensile strengths of Astroloy in 3.5- and 35- MN/m^2 hydrogen at room temperature were 95 and 90 percent of the respective values determined in helium. The notched and smooth tensile properties in hydrogen at elevated temperatures were essentially unchanged from values measured in helium (figs. 2(a) and (b)). Although the creep rate was increased and rupture life slightly decreased by hydrogen at 675°C , rupture ductility was unchanged (fig. 2(c)).

Differences in alloy microstructure, resulting from material processing or heat treatments or experimental conditions such as hydrogen pressure or impurities, were believed responsible for the differences reported for these two alloys. Hence, one purpose of this investigation was to determine whether microstructural or test procedure variations were responsible for these reported differences. To accomplish this purpose NASA contracted with Pratt & Whitney Aircraft Company and Rocketdyne to perform notched tensile tests on Udimet 700 in 3.5- MN/m^2 gaseous hydrogen.

Another purpose of this investigation was to evaluate candidate APU turbine disk alloys for resistance to hydrogen environment embrittlement. To accomplish this purpose contractors performed notched and smooth tensile tests and creep tests on Udimet 700, Astroloy, and V-57 in hydrogen over the temperature range 23° to 760° C. The alloy V-57 is one of the iron-base superalloys (stable austenitic stainless steels), a class of materials shown to have good resistance to hydrogen environment embrittlement (refs. 4 and 5). This alloy was used for the turbine disks in the APU system performance test (ref. 1) because of program scheduler requirements. However, the nickel-base superalloys would offer significant performance advantages over V-57 if they could provide reliable service in advanced APU's.

MATERIALS, SPECIMENS, AND PROCEDURE

Materials

The compositions of alloys tested in previous investigations (refs. 2 and 3) and in this investigation are given in table I. All compositions are within nominal specification ranges (refs. 6 and 7). Alloy heat or specification numbers, material forms, heat treatments, and resultant grain sizes are listed in table II. All alloys were heat treated in accordance with standard specifications (refs. 6 to 8) with the exception of some Udimet 700, which received both the standard Udimet 700 heat treatment and the standard Astroloy "yo-yo" heat treatment (see table II).

Specimens

The specimens used in this investigation are shown in figure 3. The notched tensile specimen is shown in figure 3(a) along with its dimensions and those of Udimet 700 and Astroloy specimens used in previous investigations (refs. 2 and 3). The Astroloy specimen was similar to the one of this investigation, whereas the Udimet 700 specimen was different. The smooth specimens used for tensile and creep testing of Astroloy are shown in figures 3(b) and (c). The smooth tubular specimen used for smooth tensile and creep testing of V-57 is shown in figure 3(d). This specimen was used because the contractor did not have a pressure vessel capable of hydrogen testing at elevated temperatures. The bore of this tubular specimen was pressurized with hydrogen, and the external surface was exposed to air. Notched tensile testing could not be performed with this specimen.

All Udimet 700 test specimens were machined from bar stock with their longitudinal axes oriented parallel to the rolling direction, as was done in the work of reference 2.

Specimens cut from Astroloy forgings for the investigation of reference 3 were oriented circumferentially, close to the periphery of the forging. Specimens cut from the Astroloy forgings procured for this investigation were oriented in the radial direction, which is the direction of maximum stress in the APU disks. Specimens of V-57 were oriented in both circumferential and radial directions. Prior to testing, all specimens were cleaned with trichloroethylene, dried, rinsed with acetone, dried, rinsed with methanol, and dried.

Experimental Test Procedures

Test environments. - The experimental procedures used by each investigator to conduct the mechanical tests in hydrogen (H_2) are summarized in table III. After cleaning, specimens were sealed in the test vessels, which were then evacuated and back-filled a number of times with nitrogen or hydrogen. The test vessel (or the V-57 tubular specimen) was pressurized and purged with hydrogen and then finally filled with hydrogen to the desired test pressure. Baseline tensile tests were conducted with the specimens exposed to helium (He) or air. Reported impurities in the hydrogen and helium are listed in table III. Test tensile loads were corrected for sliding seal friction loads.

Resolution of differences in the literature. - In order to resolve the differences regarding the degree of embrittlement reported for Udimet 700 and Astroloy, notched tensile specimens from one heat of Udimet 700 bar stock were machined by Lewis. Tensile tests of these specimens were then conducted by the two contractors at room temperature, 3.5-MN/m² hydrogen pressure, and a crosshead speed of 0.25 centimeter per minute (see table III). Variable test conditions between the contractors were hydrogen purity and evacuation and pressurization procedures. Notched tensile test results are listed in table IV.

Alloy evaluation. - Additional mechanical tests were conducted in hydrogen by the contractors on Udimet 700 that had been given the "yo-yo" heat treatment, on Astroloy, and on V-57. Notched tensile tests were conducted at 23° and 675° C at a crosshead speed of 0.25 centimeter per minute. Smooth tensile tests were conducted at 23°, 565°, 675°, and 760° C at a crosshead speed of 0.25 or 0.01 centimeter per minute. All of these test conditions are summarized in table III, and all tensile test results are presented in tables IV to VI.

Creep tests were performed on Astroloy and V-57 alloys at 565° and 675° C. Creep elongation and 0.2-percent total creep were measured by means of a linear transducer. Residual tensile properties of creep-exposed Astroloy and V-57 specimens were measured by conducting tensile tests at room temperature in air at a crosshead speed of 0.01 centimeter per minute. All creep test results are listed in tables VII and VIII. The reduction of area data listed in table VIII for tubular V-57 specimens are

"apparent reduction of area." These data were calculated only from changes in the outer diameter of the tubular specimens.

RESULTS AND DISCUSSION

Resolution of Differences in the Literature

One purpose of this investigation was to determine whether differences in contractors' experimental test procedures (table III) or material variations were responsible for the reported literature variations in hydrogen environment embrittlement of Udimet 700 and Astroloy. The notched tensile test results obtained in this investigation for Udimet 700 are listed in table IV together with comparative literature results for Udimet 700 and Astroloy obtained in previous investigations (shown in figs. 1 and 2). It is apparent that (1) the current results obtained by the two contractors are in excellent agreement with each other (notched tensile strengths in hydrogen, 59 and 53 percent of the baseline), and (2) the current results also agree well with those previously reported for Udimet 700 (notched tensile strength in hydrogen, 66 percent of the baseline) but not with those for Astroloy (notched tensile strengths in hydrogen, 90 and 95 percent of the baseline).

Although this investigation was not designed to ascertain conclusively the role of all experimental variables, the results obtained strongly suggest that differences in contractor experimental variables are not significant. For example, similar degrees of embrittlement were determined for Udimet 700 when tested under the following conditions: 0.45- to 0.8-centimeter specimen notch diameter, 3.5- to 50-MN/m² hydrogen pressure, liquid boiloff against bottled hydrogen, and 0.12- to 0.25-centimeter-per-minute crosshead speed. Hence, it can be concluded that variations in the alloys rather than experimental procedures were responsible for the reported differences between Astroloy and Udimet 700.

Evaluation of Nickel-Base Superalloys

Metallography of Udimet 700. - Specimens of Udimet 700 that had been tested in helium (or air) and hydrogen were examined metallographically (see fig. 4). The following observations are representative of the results obtained by both contractors: (1) the alloy had a large grain size and a large γ' precipitate size (formed during heat treating at high temperatures) together with some finer γ' (formed during heat treating at intermediate temperatures); (2) fracture in helium (or air) was intergranular except immediately below the root of the notch; and (3) fracture in hydrogen was totally intergranular and resulted in slightly more secondary cracking perpendicular to the failure plane.

The following observations were made from a metallographic comparison of this Udimet 700 (fig. 4) with the previously tested (ref. 3) Astroloy (fig. 5): (1) Udimet 700 fractured intergranularly, while Astroloy fractured transgranularly; (2) Udimet 700 had a much larger grain size (ASTM 0-4) than did Astroloy (ASTM 5-7); and (3) Udimet 700 had a larger γ' precipitate size than did Astroloy.

The larger grain size observed for Udimet 700 presumably was due to differences in processing history between Udimet 700 and Astroloy. For example, Udimet 700 was rolled to 1.9-centimeter-diameter bar stock, solution heat treated at 1175° C, and air cooled, whereas Astroloy was forged to a 4.2-centimeter-thick pancake, solution heat treated at 1105° C, and oil quenched. The actual amount of rolling or forging deformation that each alloy received is unknown. However, the higher solution-heat-treating temperature given the Udimet 700 alloy would account for the larger grain size observed in the Udimet 700. In addition, the higher aging temperature of the standard three-step aging heat treatment given the Udimet 700 alloy would account for the larger γ' precipitate size observed in the Udimet 700 than in the Astroloy, which received a "yo-yo" four-step aging heat treatment at lower temperatures designed to produce a fine γ' size.

Notched tensile testing of Udimet 700 with "yo-yo" heat treatment. - A few specimens of Udimet 700 bar stock were given a second heat treatment according to the Astroloy "yo-yo" specification (refs. 7 and 8). The notched tensile strength in helium at room temperature for this material was approximately 10 percent lower than that for Udimet 700 in the standard heat-treated condition. The notched tensile strength in hydrogen at room temperature for this material was 50 percent of its strength in helium, as compared with 53 percent for Udimet 700 in the standard heat-treated condition (see table IV).

Metallography of Udimet 700 with "yo-yo" heat treatment. - Metallographic examination of this re-heat-treated Udimet 700 bar stock revealed the following: (1) a microstructure almost identical to that discussed previously for Udimet 700 with the standard heat treatment (compare figs. 6 and 4), (2) intergranular fracture in both helium and hydrogen but to a slightly greater degree and with more secondary cracking in hydrogen than in helium, and (3) a γ' size unchanged from that of the standard Udimet 700 discussed previously. It is not surprising that this second heat treatment did not cause any significant microstructural changes in light of the 70° C lower solution annealing temperature called for in the "yo-yo" specification. The large grain size had been established in the initial solution annealing treatment and would not be expected to be altered during the subsequent heat-treating steps.

Notched tensile testing of Astroloy. - Material that met both Astroloy chemistry and "yo-yo" heat-treatment specifications was purchased from a commercial source in the form of fully heat treated 4.5-centimeter-thick pancake forgings. Notched tensile tests of this material at room temperature indicated a degradation of only 8 percent in hydrogen as compared with the baseline helium tests (see table IV). These results,

which are shown in figure 7 (see also table IV), agree well with the previously reported (ref. 3) tests on Astroloy, which showed a degradation in notched tensile strength of 4 percent in 3.5-MN/m^2 hydrogen. Two additional notched tensile tests were conducted at 675°C . These tests indicated a degradation of only 6 percent in notched tensile strength in hydrogen as compared with helium, as shown in figure 8.

Metallographic examination of Astroloy. - Examination of Astroloy specimens tested at 23°C revealed the following: (1) completely ductile transgranular fracture in the specimens tested in helium (fig. 9(a)), (2) predominantly transgranular fracture with a few indications of intergranular fracture in the specimens tested in hydrogen (fig. 9(b)), (3) a duplex grain structure consisting of large residual cast dendritic grains and finer grains heavily loaded with carbides (figs. 9(c) and (d)), and (4) uniformly sized γ' precipitates (fig. 9(e)) which were finer than those found in Udimet 700 but not quite as fine as those observed in the Astroloy tested in the previous investigation (fig. 5).

The duplex microstructure, which is indicative of a light and nonuniform forging procedure, may be responsible for the low strength of this Astroloy. The notched tensile strength in helium was about 1390 MN/m^2 , which is 20 percent lower than the strength of the Astroloy tested in the previous investigation (ref. 3).

Smooth tensile testing of Astroloy. - In addition to the notched tensile testing at 23° and 675°C , smooth tensile tests were performed on Astroloy at 23° , 565° , 675° , and 760°C to evaluate more fully the effect of hydrogen on this alloy. The results of these tensile tests are presented in figure 8 and are listed in table V. The most significant effect of hydrogen on tensile properties occurred at room temperature. Specifically, ultimate tensile strength was reduced 5 percent (from 1215 to 1155 MN/m^2) and reduction of area was reduced 20 percent (from 10 to 8 percent) by testing in hydrogen. The embrittling effect of hydrogen diminished as the tensile test temperature was increased.

Scanning electron micrographs of the smooth tensile specimens tested at room temperature are presented in figure 10. No significant differences between fracture modes in helium and hydrogen were detectable (compare figs. 10(a) and (b)).

Creep testing of Astroloy. - In the two creep tests at 565°C hydrogen had no detrimental effect on either the creep properties (fig. 11 and table VII) or the postcreep residual tensile properties at room temperature (table VII). The two creep tests conducted at 675°C did not yield any conclusive result as to the effect of hydrogen on the creep properties of the alloy because the specimen tested in hydrogen failed at one of the extensometer clamp ridges, and hence, the test could not be considered valid. In spite of the location of this failure the test data determined in this investigation suggest that Astroloy is not a notch-sensitive material in either hydrogen or helium. The data listed in tables IV and V indicate that the ratio of notched to smooth tensile strength in helium at room temperature for Astroloy was 1.15. The ratio of notched strength in hydrogen to smooth baseline tensile strength at 23°C was approximately 1.03. Obviously,

additional creep test data are required for a conclusive statement as to the effect of gaseous hydrogen on the creep properties of Astroloy.

Evaluation of V-57 Iron-Base Superalloy

As mentioned in the INTRODUCTION, V-57 was used for the turbine disks for the experimental APU. It was selected for this purpose because it is one of the iron-base superalloys (stable austenitic stainless steels), a class of materials known to be relatively resistant to hydrogen environment embrittlement as well as other types of hydrogen embrittlement (refs. 3 to 5). The resistance of austenitic stainless steels to hydrogen environment embrittlement is typified by the alloy A-286, for which there are more test data. The higher strength of V-57 relative to A-286 was necessary for the APU application.

Smooth tensile testing. - Tensile tests were conducted with tubular V-57 specimens at 23⁰, 565⁰, and 675⁰ C. The results shown in table VI indicate that hydrogen at 1.7 MN/m² had no embrittling effect on V-57. All tensile properties, ultimate tensile strengths, apparent reductions of area, and elongations were essentially equivalent in both the baseline helium tests and those conducted in hydrogen.

These results demonstrating the high resistance of V-57 to hydrogen environment embrittlement are in agreement with previously reported investigations on A-286 (ref. 3) and V-57 (unpublished data obtained from A. W. Thompson and W. T. Chandler of Rocketdyne) conducted at hydrogen pressures of 35 and 70 MN/m², respectively. The results shown in table VI demonstrate that hydrogen does not affect either the smooth or notched tensile properties of these austenitic stainless steels over the temperature range 23⁰ to 675⁰ C.

Creep testing. - A series of tests was conducted to determine the influence of hydrogen on the creep properties of V-57. Test results are listed in table VIII and are plotted in figure 12 together with comparative handbook data (ref. 6). Tests conducted in helium in this investigation to determine both the 0.2-percent creep strength and the rupture strength of the alloy agreed well with handbook values.

Creep tests conducted in hydrogen at 565⁰ C indicated that 0.2-percent creep elongation of both radially and circumferentially oriented specimens was degraded approximately 0.8 Larson-Miller parameter from baseline values. One radially and one circumferentially oriented specimen failed during testing in hydrogen about 2 Larson-Miller parameters lower than the baseline. These specimens had measured rupture elongations of only 2 percent in hydrogen as compared with 12 percent for rupture elongation in helium. The results of metallographic examinations of these prematurely failed specimens are discussed in the next section.

Creep tests conducted in hydrogen at 675^o C indicated that 0.2-percent creep occurred at approximately 0.6 Larson-Miller parameter less than that of the helium baseline for circumferentially oriented specimens. No degradation in 0.2-percent creep life was determined for radially oriented specimens at this higher creep testing temperature. Creep testing in hydrogen also resulted in an acceleration of creep elongation of V-57 at both 565^o and 675^o C, as shown in figure 13. Specifically, when tested under identical creep conditions, specimens tested in hydrogen had creep rates significantly greater than those of specimens tested in helium. A similar influence of hydrogen is reported in reference 3 for another austenitic stainless steel, A-286, and for Astroloy (see fig. 2(c)). Neither A-286 nor Astroloy suffered any loss of rupture ductility. Although a rationalization for hydrogen-accelerated creep was not pursued in either this investigation or that of reference 3, the authors suggest that hydrogen may accelerate the initial stages of creep deformation by promoting dislocation motion near the surfaces of test specimens.

Metallographic examination of creep-ruptured specimens. - As noted in the discussion regarding figure 12, two specimens failed prematurely when creep tested in hydrogen. The results of metallographic examination of these specimens indicated that a hydrogen-induced crack originated at or near the inner diameter and propagated through the wall of the internally pressurized tubular specimen. Figure 14 shows the fracture surface of one of the specimens. Apparently, after the crack penetrated the specimen wall, it spread laterally until a ductile overload shear failure occurred. The main hydrogen-induced fracture zone was almost totally intergranular, while the shear lip was transgranular.

Postcreep mechanical properties. - Tensile tests were performed at room temperature in air on all creep-exposed specimens of V-57 to determine residual strength and ductility. The results, which are shown in figure 15 and listed in table VIII, indicate that long-term creep exposure in hydrogen resulted in subsequent embrittlement during tensile testing. Generally, the ductility of specimens creep-exposed in hydrogen was about 40 percent less than that of specimens creep-exposed in helium. Tensile strengths also were reduced slightly by creep exposure in hydrogen.

It should be emphasized that these tensile tests were conducted in air. Therefore, embrittlement was due to hydrogen that had been absorbed within the specimens during the prior creep test in hydrogen. Chemical analysis of selected specimens confirmed absorption of hydrogen from a baseline of 4 parts per million to as high as 16 parts per million (see table VIII). It should also be noted that these tensile tests were conducted at conditions chosen to maximize the embrittling effect of hydrogen (room temperature and a low test speed of 0.01 cm/min). This type of embrittlement is termed internal reversible hydrogen embrittlement (ref. 5).

Metallographic examination of creep-exposed specimens. - The effect of absorbed hydrogen is also evident from an examination of specimen fracture surfaces (fig. 16). A specimen creep-exposed in helium which exhibited 15-percent tensile elongation during subsequent testing at room temperature is shown in figure 16(a). The fracture mode of this specimen was predominantly transgranular and only slightly intergranular. A specimen creep-exposed in hydrogen which exhibited only 4-percent tensile elongation during subsequent testing at room temperature is shown in figure 16(b). In contrast to the specimen shown in figure 16(a), the fracture mode of this specimen consisted of approximately equal amounts of transgranular and intergranular fracture.

It should be emphasized that the respective fracture modes of each of these specimens was uniform throughout the entire fracture surface, which indicated uniform absorption of, and embrittlement by, hydrogen. These fractures were in direct contrast to those observed for the two specimens that failed prematurely during creep testing in hydrogen (fig. 14), which failed by localized hydrogen embrittlement, crack initiation and propagation, and ductile overload of the remaining cross section.

CONCLUDING REMARKS

Both objectives of this investigation were fulfilled: microstructural variations between Udimet 700 and Astroloy rather than variations in test procedures were determined to be responsible for the reported differences in susceptibility to hydrogen environment embrittlement, and both Astroloy and V-57 were evaluated for use as turbine disks for advanced APU's in terms of their resistance to hydrogen embrittlement.

This investigation confirmed previous results (ref. 2) that Udimet 700 bar stock is severely embrittled when mechanically tested in gaseous hydrogen. This extreme sensitivity to embrittlement of Udimet 700 is presumably related to its microstructure. The high solution annealing temperature specified for this alloy results in a large grain size, while the conventional precipitation annealing temperatures used to age this alloy result in a large γ' precipitate size.

This investigation also confirmed previous results (ref. 3) that Astroloy forgings exhibit a high degree of resistance to hydrogen environment embrittlement during short-term testing and possibly during long-term testing (table IX). Therefore, this alloy could be considered for use as the turbine disk alloy for advanced versions of the APU and, if used, would permit a higher turbine inlet temperature and/or a higher rotational speed than would be possible with V-57. Specific propellant consumption could then be reduced by about 10 percent.

The resistance exhibited by Astroloy forgings to embrittlement by hydrogen is also presumably due to its microstructure, which results from forging and its special heat-treatment schedule. The alloy microstructure consists of fine grains and a fine γ'

precipitate. The beneficial effect of fine grain size on the resistance of Inconel 718 to hydrogen environment embrittlement had been reported previously (ref. 4). Although the beneficial effect of decreasing the γ' precipitate size on the resistance of Rene 41 to internal reversible hydrogen embrittlement had been demonstrated (ref. 9), no such effect had been previously reported for hydrogen environment embrittlement.

The alloy V-57 is one of the iron-base superalloys (stable austenitic stainless steels), a class of materials generally quite resistant to hydrogen environment embrittlement (refs. 4 and 5). The results of this investigation demonstrated the good resistance of this alloy to embrittlement only during short-term tensile testing (table IX). Significant reductions in creep and rupture lives, as well as postcreep residual ductility, were determined in this investigation. Despite these laboratory results, V-57 turbine disks successfully completed short-time performance testing in the experimental APU (ref. 1).

Although the subject is not developed in this report, these data are applicable not only to aerospace propulsion and power systems, but also to terrestrial systems involving transmission and generation of energy. A notable example is the proposed electric-energy-producing utility powerplant, wherein hydrogen is reacted in a single combustor to produce superheated steam. The massive boiler necessary in commercial steam systems is eliminated, and turbine inlet temperatures can be increased to particularly high levels when hydrogen is used as a turbine metal coolant. Potential powerplant system efficiencies of 45 to 55 percent have been estimated for such a concept. In addition, environmental pollution would be very low or even nonexistent. Hydrogen is also vital to the production of plastics and fertilizers for consumer use. The efficiency of hydrogen production could be increased if higher temperatures and pressures could be used safely. Hence, the need for structural alloys which are resistant to degradation by hydrogen will become increasingly important in the future.

SUMMARY OF RESULTS

One purpose of this investigation was to resolve the differences reported in the literature regarding the degree of hydrogen environment embrittlement reported for Udimet 700 and Astroloy. Another purpose was to evaluate candidate superalloys for resistance to embrittlement so that a superalloy could be selected for reliable service for the turbine disks of an advanced Space Shuttle auxiliary power unit (APU). Udimet 700 and Astroloy (nickel-base superalloys) and V-57 (an austenitic iron-base superalloy) were mechanically tested in gaseous hydrogen at pressures of 3.5 and 1.7 MN/m², respectively. Notched and smooth tensile tests were conducted over the temperature range 23° to 760° C. Creep tests were conducted at 565° and 675° C, and postcreep tensile tests were conducted in air at room temperature to determine residual ductility

and strength. The following results were obtained:

1. The reported differences regarding the degree of susceptibility to hydrogen environment embrittlement of Udimet 700 and Astroloy were resolved as being due to microstructural variations rather than experimental testing variations.

2. Udimet 700 bar stock was confirmed to be extremely susceptible to embrittlement when mechanically tested in hydrogen. Notched tensile tests of Udimet 700 at room temperature demonstrated embrittlement to levels of about 50 percent of baseline values in air.

3. Astroloy forgings were confirmed to have a high degree of immunity to embrittlement by hydrogen. Both smooth and notched tensile strengths in hydrogen were at least 92 percent of values determined in air over the temperature range 23° to 760° C. Limited creep test data for Astroloy did not indicate degradation due to hydrogen.

4. The alloy V-57 was not embrittled during smooth tensile testing over the temperature range 23° to 675° C. However, creep and rupture testing at 565° and 675° C indicated degradations of approximately 1 to 2 Larson-Miller parameters. Postcreep tensile testing at room temperature revealed that residual ductility of V-57 was lowered by approximately 40 percent by creep testing in hydrogen.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, July 22, 1975,
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TABLE 1. - ALLOY COMPOSITIONS

Alloy	Refer- ence	Specification or heat number	Nickel	Iron	Chrom- ium	Cobalt	Aluminum	Titanium	Molyb- denum	Boron	Vana- dium	Silicon	Manga- nese	Carbon
Concentration, wt. %														
Udimet 700	7	Nominal composition	Bal.	0-4	14-16	17-20	3.75-4.75	2.75-3.75	4.5-6.0	0.025-0.035	----	----	----	0.03-0.10
Astroloy	7.8	Nominal composition	Bal.	0-0.5	14-16	16-18	3.85-4.15	3.3-3.7	4.5-5.5	0.025-0.035	----	----	----	0.04-0.10
Udimet 700	2	Pratt & Whitney Aircraft	Bal.	0.25	15.0	16.7	4.0	3.52	5.0	.022	----	0.1	0.10	.08
Astroloy	3	Specification 1019 Pratt & Whitney Aircraft specification 1013; heat LKCC	Bal.	.15	15.1	16.65	3.99	3.31	5.3	.025	----	.05	.015	.08
Udimet 700	This investi- gation	Special Metals heat 8-1995	Bal.	.19	14.6	19.0	4.46	3.38	4.85	.027	----	<.1	<.10	.07
Astroloy	This investi- gation	Special Metals heat 8-2963	Bal.	.3	15.2	17.7	4.1	3.5	4.9	.026	----	.05	.12	.05
V-57	6	Nominal composition	25.5-28.5	Bal.	13-16	-----	0.1-0.35	2.7-3.2	1-1.5	0.005-0.025	0-0.5	0-.75	0-.35	0-.08
V-57	This investi- gation	Carpenter order M45809	26.07	Bal.	13.76	-----	.28	3.09	1.17	.01	.23	.20	.14	.027

TABLE II. - FORMS, HEAT TREATMENTS, AND GRAIN SIZES OF ALLOYS TESTED

Alloy	Refer- ence	Form	Heat treatment				Grain size, ASTM number	Specimen preparation
			Temperature		Time, hr	Cooling (a)		
			°C	°F				
Udimet 700	2	1.6-cm- (0.63-in. -) diam bar	1175 1080 845 760	2150 1975 1550 1400	4 5 24 16	AC AC AC AC } (b)	Large	-----
Astroloy	3	45-cm- (17-6. -in. -) diam by 4.2-cm- (1.65-in. -) thick pancake forging	1105 870 980 650 760	2025 1600 1800 1200 1400	4 8 4 24 8	OQ AC AC AC AC } (c)	5-7, occa- sionally 4	Machined from fully heat treated material
Udimet 700	This investi- gation	1.9-cm- (0.75-in. -) thick bar	(b)	(b)	(b)	(b)	0-4	Roughly machined after 1080° C (1975° F) heat- ing; finally machined from fully heat treated material
Udimet 700	This investi- gation	1.9-cm- (0.75-in. -) thick bar	(b), (c)	(b), (c)	(b), (c)	(b), (c)	0-5	Roughly machined after 1080° C (1975° F) heat- ing; finally machined from fully heat treated material
Astroloy	This investi- gation	23-cm- (9-in. -) diam by 4.5-cm- (1.75-in. -) thick forging	(c)	(c)	(c)	(c)	3-5, with very fine duplex structure	Machined from fully heat treated material
V-57	This investi- gation	18-cm- (7-in. -) diam by 4.5-cm- (1.75-in. -) thick forging	1010 815 730	1850 1500 1350	2 1 16	OQ AC AC	3-5	Roughly machined after 1010° C (1850° F) heat- ing; finally machined from fully heat treated material

^aAir cooled, AC; oil quenched, OQ.

^bStandard heat treatment.

^c"Yo-yo" heat treatment.

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TABLE III - EXPERIMENTAL TEST CONDITIONS

Hydrogen source	Reference	Impurities in hydrogen or helium, ppm						Evacuation pressure, N m^{-2}	Backfill, cm^3		Hydrogen purity, cm^3		Hydrogen pressure, cm^2	Tensile test						Creep test, alloy tested	
		Oxygen		Nitrogen		Carbon dioxide			Water	Gas	Pressure, MN m^{-2}	Cycles		Pressure, MN m^{-2}	Alloy tested	Notched		Smooth			
		C ₁	C ₂	C ₃	C ₄	C ₅	C ₆									Crosshead speed, cm min^{-1}	Crosshead speed, cm min^{-1}	Alloy tested	Crosshead speed, in. min^{-1}		
Liquid boil off	2	b, 0.5	b, 1	b, 0.2	b, 1	b, 1	---	13	Hydrogen	14	2000	2	---	52	Udimet 700	0.25	0.1	---	---		
Liquid	3	b, 1	---	---	---	---	---	13	---	---	---	6	3.5 or 35	500 or 5000	Astroloy	0.12	0.05	---	---		
Liquid	This investigation, contractor 1	b, 0.1	---	---	---	---	---	7	Nitrogen twice	6	50	3	3.5	500	Udimet 700, Astroloy	0.25	0.1	Astroloy	0.25	0.1	Astroloy
Bottle hydrogen, 99.999 percent pure plus oxygenic absorption	This investigation, contractor 2	b, 0.5, 0.7, C ₁ , C ₂	b, 0.8, 3.4, C ₃ , C ₄	b, 0.5, C ₅ , C ₆	b, 1	b, 1	b, 1	2.7	Hydrogen six times	1.7 or 3.5	250 or 500	3	3.5 or 9.3	500 or 50	Udimet 700, Udimet 700 with standard and "Voxal" heat treatments, Astroloy	0.25	0.1	V-57	0.01	0.005	V-57

^aHelium used for some baseline tests.

^bHydrogen.

^cHelium.

TABLE IV. - NOTCHED TENSILE TEST RESULTS FOR NICKEL-BASE

SUPERALLOYS AT 23° C (73° F)

Alloy	Reference	Gas	Pressure		Notched tensile strength		Strength ratio, H ₂ /He or air
			MN/m ²	psi	MN m ²	ksi	
Udimet 700	2	Air	0.1	15	1460	212	} 0.66
		H ₂	52	7500	940	136	
		H ₂	52	7500	1000	145	
Astroloy	3	He	3.5	500	1745	253	} 0.95
		He	35	5000	1745	253	
		H ₂	3.5	500	1655	240	
		H ₂	3.5	500	1675	243	} .90
		H ₂	35	5000	1560	226	
		H ₂	35	5000	1565	227	
Udimet 700	This inves- tigation, contractor 1	He	3.5	500	1655	240	} 0.59
		He	3.5	500	1660	241	
		H ₂	3.5	500	940	136	
		H ₂	3.5	500	1005	146	
Udimet 700	This inves- tigation, contractor 2	Air	0.1	15	1680	244	} 0.53
		Air	.1	15	1705	247	
		H ₂	3.5	500	815	118	
		H ₂	3.5	500	995	144	
Udimet 700 with standard and 'yo-yo' heat treatments	This inves- tigation	He	3.5	500	1495	217	} 0.50
		He	3.5	500	1505	218	
		H ₂	3.5	500	730	106	
		H ₂	3.5	500	765	111	
Astroloy	This inves- tigation	He	3.5	500	1380	200	} 0.92
		He	3.5	500	1395	202	
		H ₂	3.5	500	1225	178	
		H ₂	3.5	500	1315	191	

TABLE V. - TENSILE TEST RESULTS FOR ASTROLOY

Specimen	Gas	Pressure		Test temperature		Strength		Reduction of area, percent	Elongation, percent
		MN/m ²	psi	°C	°F	MN/m ²	ksi		
Smooth	Air	0.1	15	23	73	1220	177	11	9
	He	3.5	500	23	73	1215	176	10	9
	H ₂	3.5	500	23	73	1095	159	7	5
	H ₂	3.5	500	23	73	1220	177	9	^a 7
	He	3.5	500	565	1050	1165	169	10	8
	H ₂	3.5	500	565	1050	1095	159	9	7
	Air	.1	15	675	1250	1270	184	17	15
	He	3.5	500	675	1250	1150	167	18	14
	H ₂	3.5	500	675	1250	1095	159	21	18
	Air	.1	15	760	1400	1015	147	6	^b 5
	He	3.5	500	760	1400	1060	154	18	12
	H ₂	3.5	500	760	1400	1075	156	19	15
Notched	He	3.5	500	675	1250	1250	181	--	--
	H ₂	3.5	500	675	1250	1170	170	--	--

^aSlow crosshead speed (0.01 cm/min).^bFailed at extensometer clamp ridge.

TABLE VI. - TENSILE TEST RESULTS FOR IRON-BASE SUPERALLOYS

Alloy	Reference	Specimen	Gas	Pressure		Test temperature		Strength		Reduction of area, percent	Elongation, percent
				MN/m ²	psi	°C	°F	MN/m ²	ksi		
A-286	^a 3	Smooth	He	35	5 000	23	73	1050	152	28	47
			H ₂	35	5 000	23	73	1075	156	28	46
			He	35	5 000	675	1250	835	121	53	27
			H ₂	35	5 000	675	1250	785	114	46	23
		Notched	He	35	5 000	23	73	1545	224	--	--
			H ₂	35	5 000	23	73	1550	225	--	--
			He	35	5 000	675	1250	1200	174	--	--
			H ₂	35	5 000	675	1250	1250	181	--	--
V-57	(b)	Smooth	He	70	10 000	23	73	1130	164	51	32
			H ₂	70	10 000	23	73	1160	168	49	32
	This investigation ^{c,d}	Smooth	Air	0.1	15	23	73	1235	179	22	15
			He	1.7	250	23	73	1255	182	25	16
			H ₂	↓	↓	23	73	1250	181	21	16
			He	↓	↓	565	1050	1000	145	30	12
			H ₂	↓	↓	565	1050	1010	146	28	11
			He	↓	↓	675	1250	850	123	23	12
			H ₂	↓	↓	675	1250	805	117	30	13

^aTest results are averages for two or three tests.^bUnpublished data from A. W. Thompson and W. T. Chandler of Rocketdyne.^cRadially oriented specimens.^dApparent reduction of area, tubular specimens.

TABLE VII. - CREEP TEST RESULTS FOR ASTROLOY

[Gas pressure, 3.5 MN m² (500 psi).]

Creep test conditions						Time for 0.2-percent total creep, hr	Postcreep tensile results in air at 23 ^o C (73 ^o F)			
Gas	Temperature		Stress		Time, hr		Tensile strength		Reduction of area, percent	Elongation, percent
	^o C	^o F	MN/m ²	ksi			MN/m ²	ksi		
He	565	1050	795	115	120	^a >120	1180	171	9	6
H ₂	565	1050	795	115	117	^a >117	1125	163	9	4
He	675	1250	690	100	64.2	58.0	1170	170	9	5
H ₂	675	1250	690	100	^b 10.6	10.4	----	---	-	-
He ^c	675	1250	795	115	87.8	-----	----	---	-	-
H ₂ ^c	675	1250	795	115	59.6	-----	----	---	-	-

^aLess than 0.1-percent creep when test was terminated.^bCreep failure at extensometer clamp ridge; no measurable rupture ductility.^cFrom ref. 3 (35-MN m² (5000-psi) gas pressure).

TABLE VIII. - CREEP TEST RESULTS FOR V-57

[Gas pressure, 1.7 MN/m² (250 psi).]

Specimen orientation	Creep test conditions						Time for 0.2-percent total creep, hr	Postcreep tensile results in air at 23 ^o C (73 ^o F)				
	Gas	Temperature		Stress		Time, hr		Tensile strength		Apparent reduction of area, percent	Elongation, percent	Concentration of H, ppm
		^o C	^o F	MN/m ²	ksi			MN/m ²	ksi			
Circumferential	Air	620	1150	585	85	112	---	----	---	--	^a 12	--
Radial	He	565	1050	620	90	^b 66	---	1225	178	12	5	--
	H ₂	565	1050	675	98	12	11	----	---	8	^a 2	--
	H ₂	565	1050	640	93	133	41	1200	174	10	4	--
Circumferential	He	565	1050	620	90	139	210	1310	190	21	14	--
	H ₂	565	1050	620	90	167	90	1255	182	11	5	--
	H ₂	565	1050	675	98	32	12	----	---	5	^a 2	--
	H ₂	565	1050	675	98	^b 39	---	1270	184	12	5	--
Radial	He	675	1250	425	62	43	11	1255	182	24	15	3
	H ₂	675	1250	360	52	113	94	1205	175	15	8	--
	H ₂	675	1250	370	54	90	94	1195	173	13	8	--
	H ₂	675	1250	380	55	65	58	1255	182	7	4	11
Circumferential	He	675	1250	345	50	162	122	1255	182	18	9	4
	H ₂	675	1250	345	50	88	60	1130	164	9	4	16
	H ₂	675	1250	345	50	64	40	1205	175	13	6	--
	H ₂	675	1250	380	55	51	26	1125	163	13	6	--

^aCreep failure; ductility values are creep-rupture results.^bErratic temperature control between test initiation and second-stage creep.

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TABLE IX. - SUMMARY OF TEST RESULTS OBTAINED IN THIS INVESTIGATION

Alloy	Heat treatment	Specimen	Notched tensile strength ratio, ^a		Ratios of smooth tensile test results, H ₂ , He or air						Ratios of creep test results, H ₂ , He or air			
			H ₂ , He or air		Ultimate tensile strength	Reduction of area	Ultimate tensile strength	Reduction of area	Ultimate tensile strength	Reduction of area	Time for 0.2-per-cent creep	Postcreep reduction of area	Time for 0.2-per-cent creep	Postcreep reduction of area
			23° C (73° F)	675° C (1250° F)										
Udimet 700	Standard	Notched	0.59	---	---	---	---	---	---	---	---	---	---	---
Udimet 700	Standard	Notched	.53	---	---	---	---	---	---	---	---	---	---	---
Udimet 700	Standard plus "Vo-yo"	Notched	.50	---	---	---	---	---	---	---	---	---	---	---
Astraloy	"Vo-yo"	Notched	.92	0.94	---	---	---	---	---	---	---	---	---	---
Astraloy	"Vo-yo"	Smooth	---	---	0.95	0.80	0.94	0.90	0.95	~1.0	1.0	1.0	0.65	---
V-57	Standard	Tubular	---	---	1.0	.84	1.0	.93	.95	~1.0	.60	.60	0.58	---

^aSpecimen failed at extensometer clamp ridge.

^bAverage of both radially and circumferentially oriented specimens.

^cTwo specimens that failed approximately 2 Larson-Miller parameters prematurely not considered.

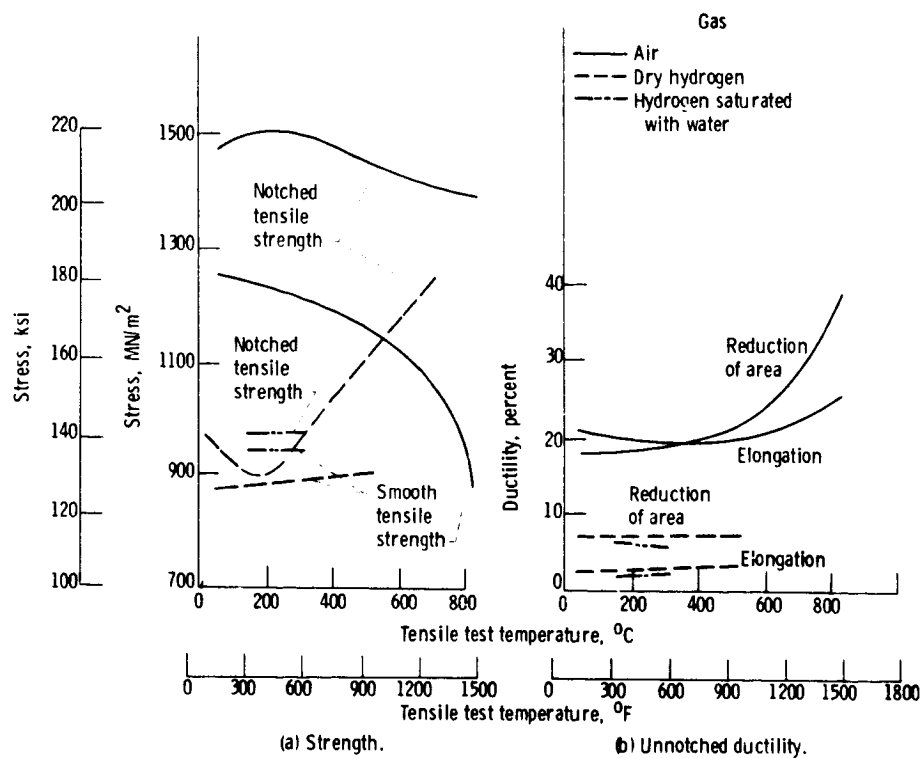


Figure 1. - Effect of test temperature on tensile properties of Udimet 700 at hydrogen pressure of 31 to 52 MN/m² (4500 to 7500 psi). Notched specimens; stress concentration factor $K_t = 8$ (ref. 2).

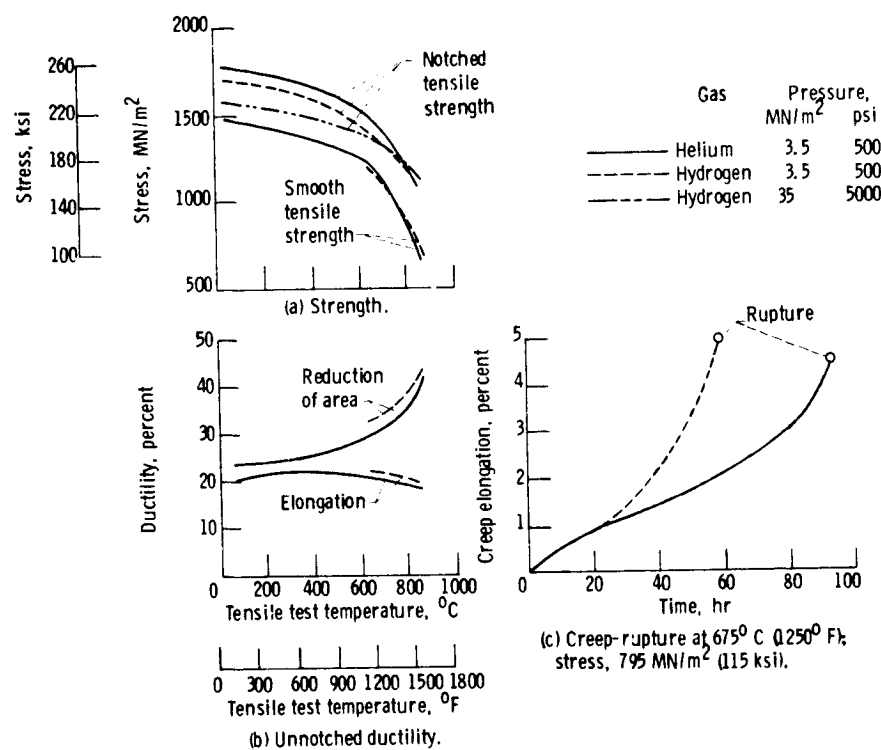
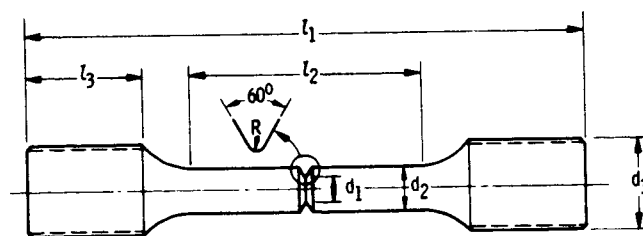
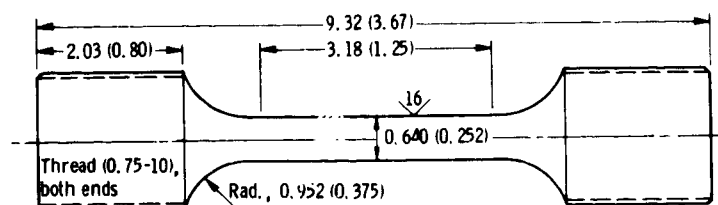


Figure 2. - Mechanical properties of Astroloy in hydrogen at pressures of 3.5 to 35 MN/m² (500 to 5000 psi) (ref. 3).

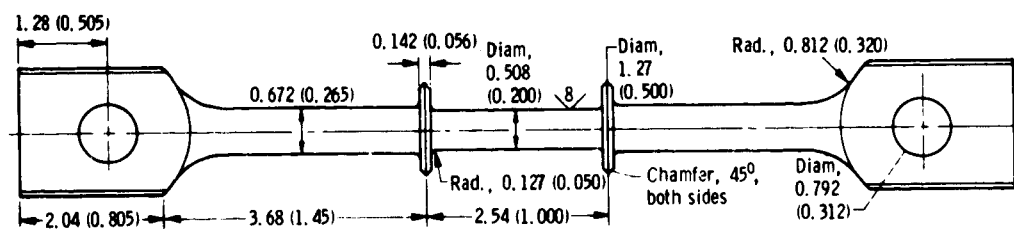


Dimension	Udimet 700 (ref. 2)		Astroloy (ref. 3) and alloys used in this investigation	
	cm	in.	cm	in.
d ₁	0.450±0.008	0.177±0.003	0.800±0.003	0.315±0.001
d ₂	0.640±0.013	0.252±0.005	1.273±0.003	0.501±0.001
d ₃	1.242-1.27	0.489-0.500	1.905	0.750
l ₁	7.62	3.00	12.09±0.03	4.76±0.01
l ₂	3.17	1.25	5.23±0.03	2.06±0.01
l ₃	1.588	0.625	2.54	1.00
Notch radius, R	0.0013 (max)	0.0005	0.005	0.002

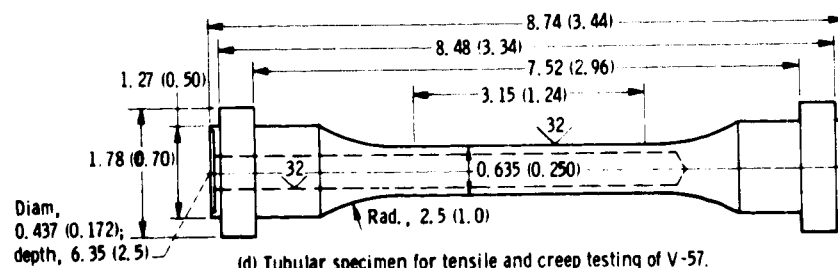
(a) Notched tensile specimen; stress concentration factor K_t, 8 to 9.



(b) Smooth tensile specimen for Astroloy.

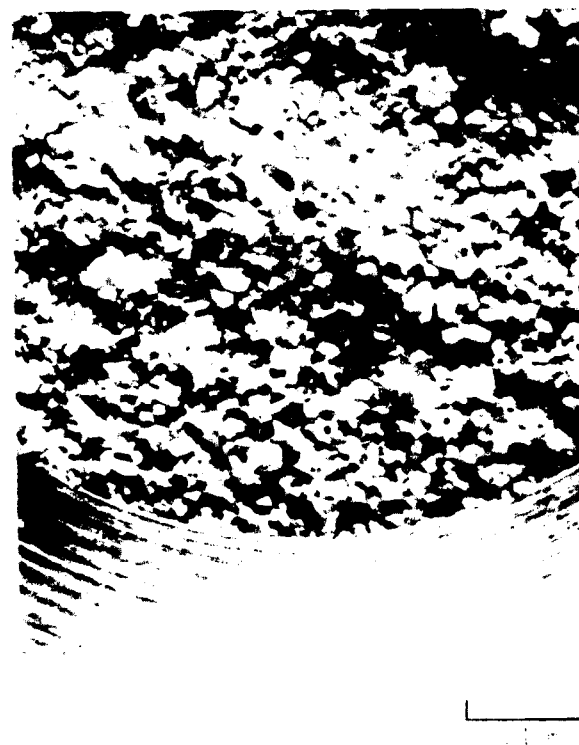
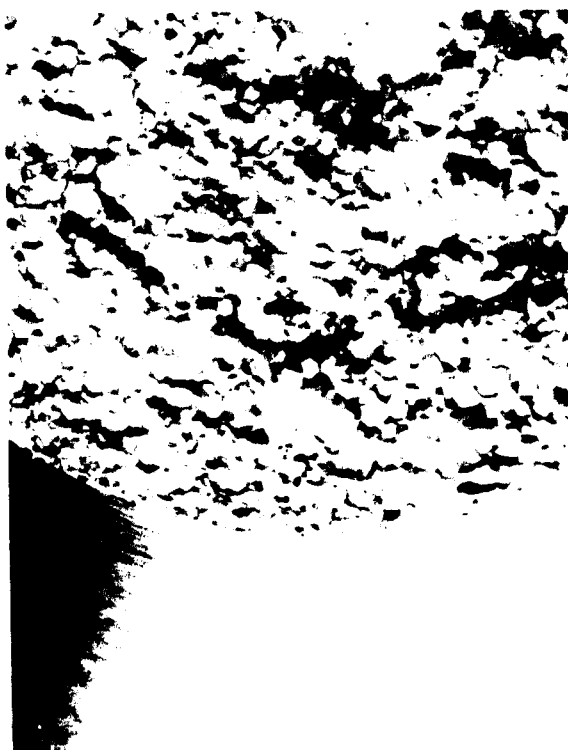


(c) Flat end creep specimen for Astroloy.



(d) Tubular specimen for tensile and creep testing of V-57.

Figure 3. - Specimens used in this investigation and those of references 2 and 3. (Dimensions in centimeters (in.)).

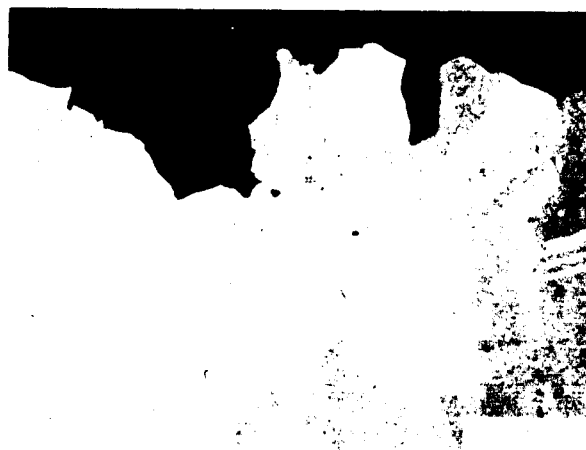
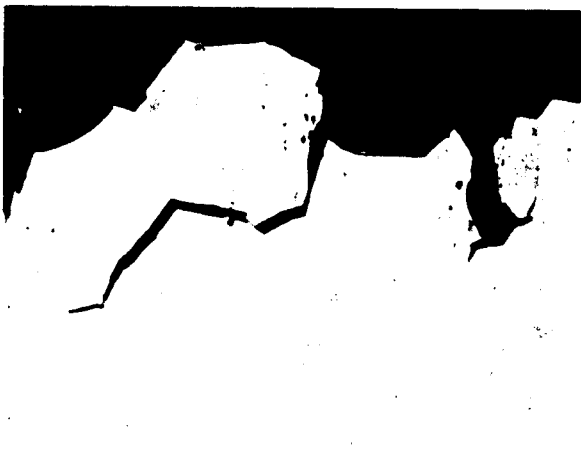
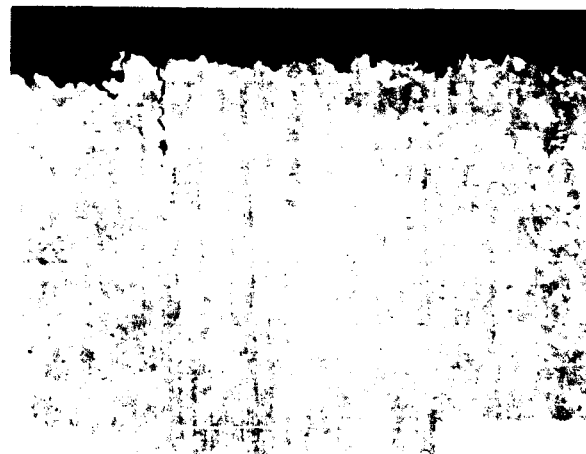
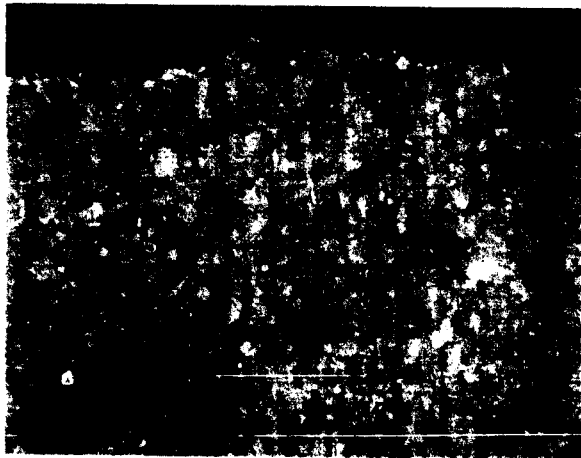


(a) Tested in air; notched tensile strength, 1705 MN/m^2 (247 ksi)

(b) Tested in hydrogen; notched tensile strength, 995 MN/m^2 (144 ksi)

Figure 1. - Photomicrographs of Udar at 700 given notched tensile test in 0.1-MN/m^2 (15-psi) air, 3.5-MN/m^2 (500-psi) hydrogen, or 3.5-MN/m^2 (500-psi) hydrogen at 23°C (73°F).

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(a) Tested in helium; matched tensile strength: 1660 MN/m^2 (241 ksi)

(b) Tested in hydrogen; matched tensile strength: 1305 MN/m^2 (116 ksi)

Figure 4. (continued)

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(e) Tested in hydrogen; notched tensile strength, 1005 MN/m^2 (146 ksi).

Figure 4. - Concluded.

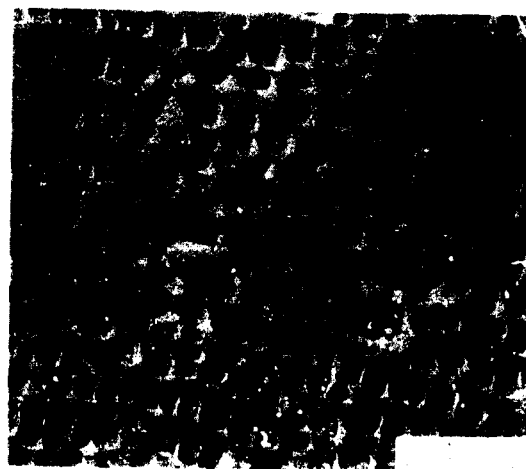
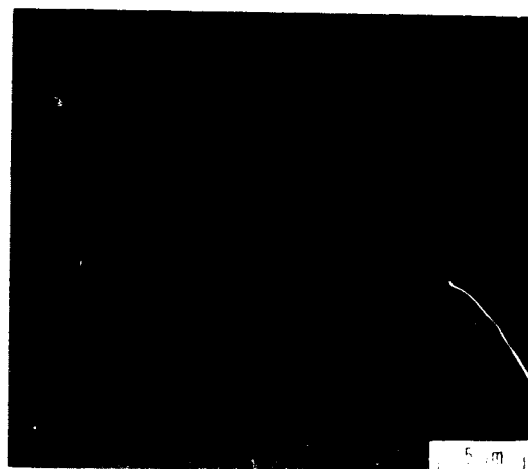
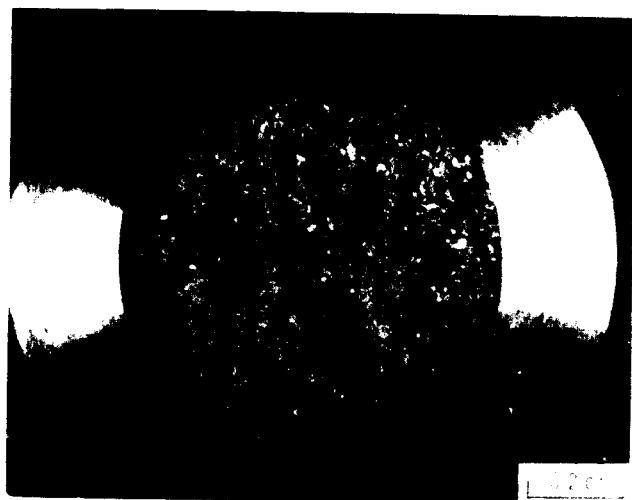
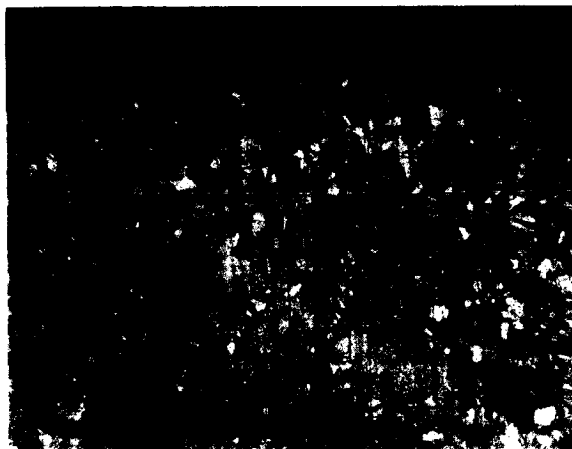


Figure 5 - Photomicrographs of Astrolloy tested in 35 MN/m^2 (5000 psi) hydrogen at 23°C (73°F). Notched tensile strength = 555 MN/m^2 (227 psi) (ref. 3).

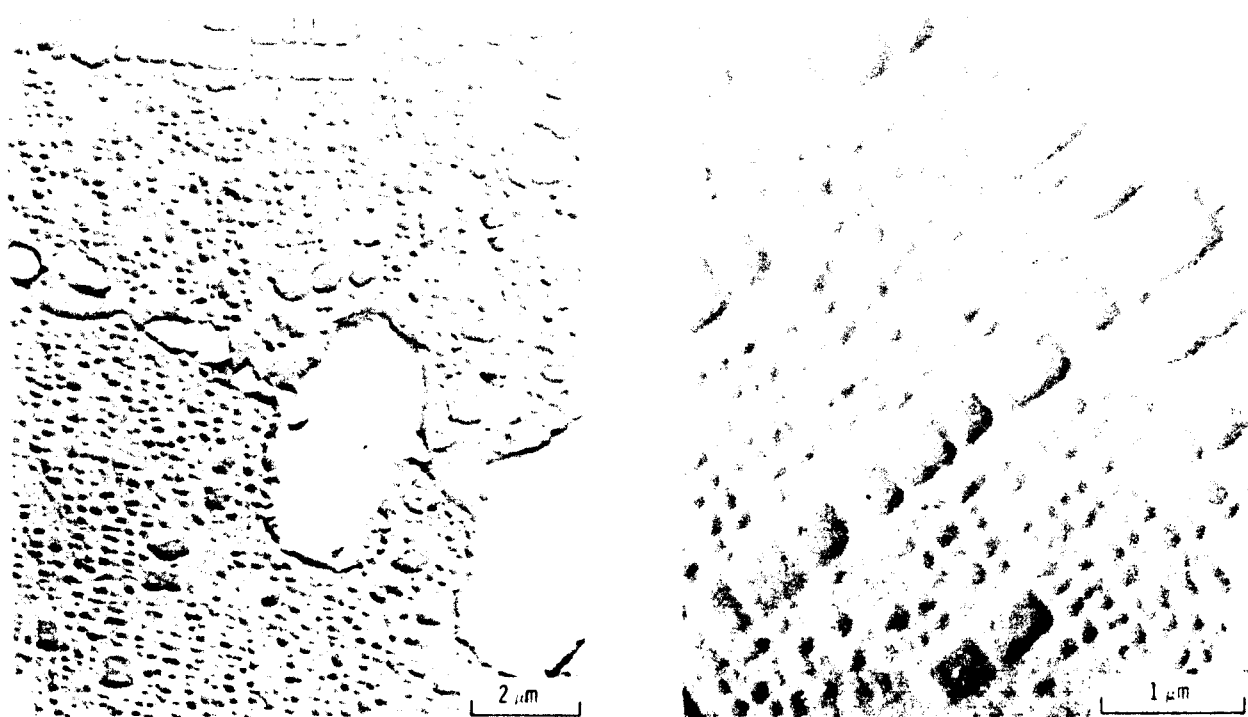
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(a) Tested in helium; notched tensile strength, 1495 MN m^{-2} (217 psi)

(b) Tested in hydrogen; notched tensile strength, 730 MN m^{-2} (106 psi)

Figure 6 - Photomicrographs of Idimet 700 given standard and "yo-yo" heat treatments and notched tensile test in 3.5 MN m^{-2} (500 psi) helium or hydrogen at 23°C (73°F)



(c) Tested in hydrogen; notched tensile strength, 730 MN m^{-2} (106 ksi).

Figure 6. - Concluded.

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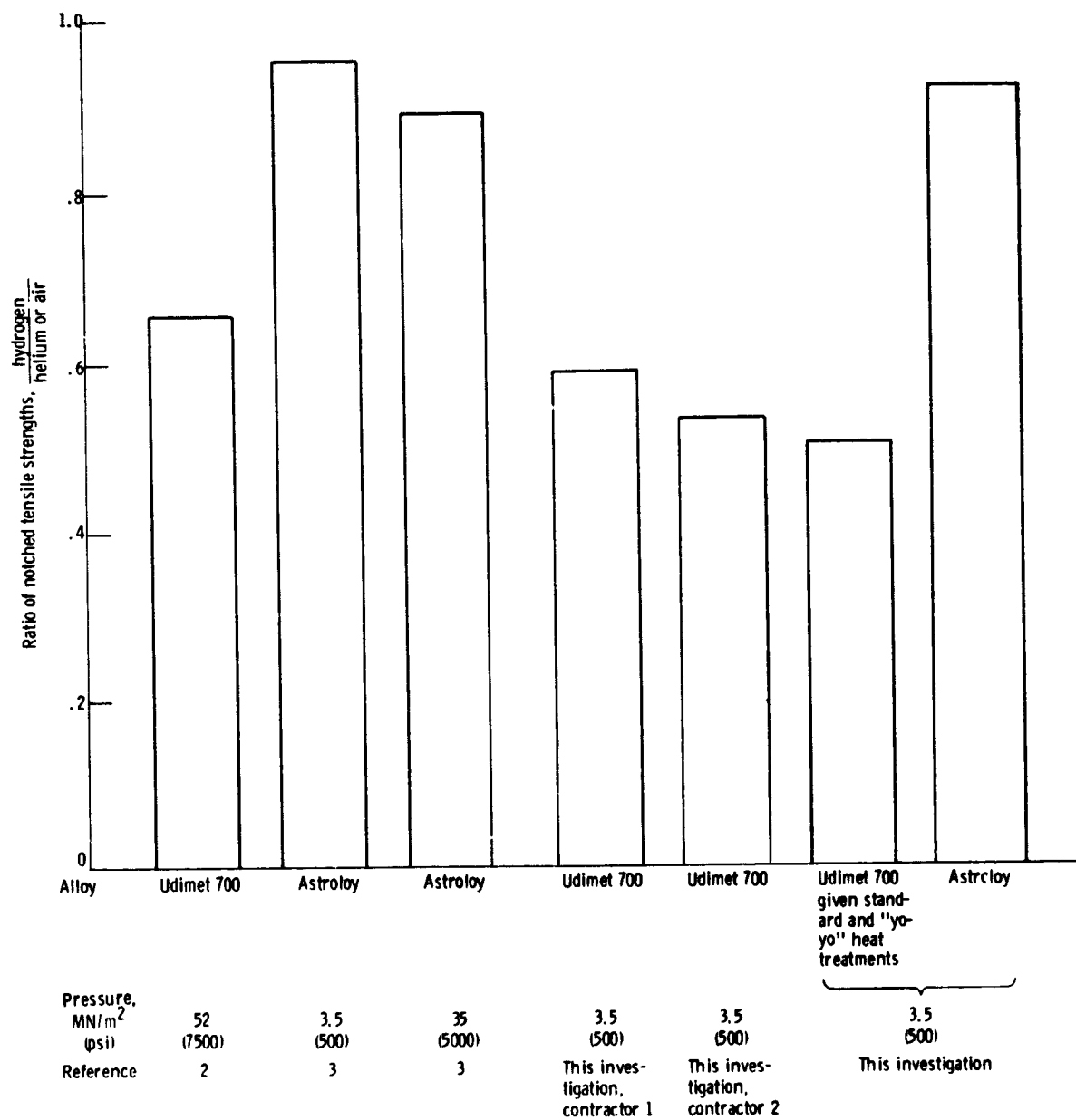


Figure 7. - Notched tensile strength results at 23° C (73° F) for Udimet 700 and Astroloy from literature (refs. 2 and 3) and this investigation.

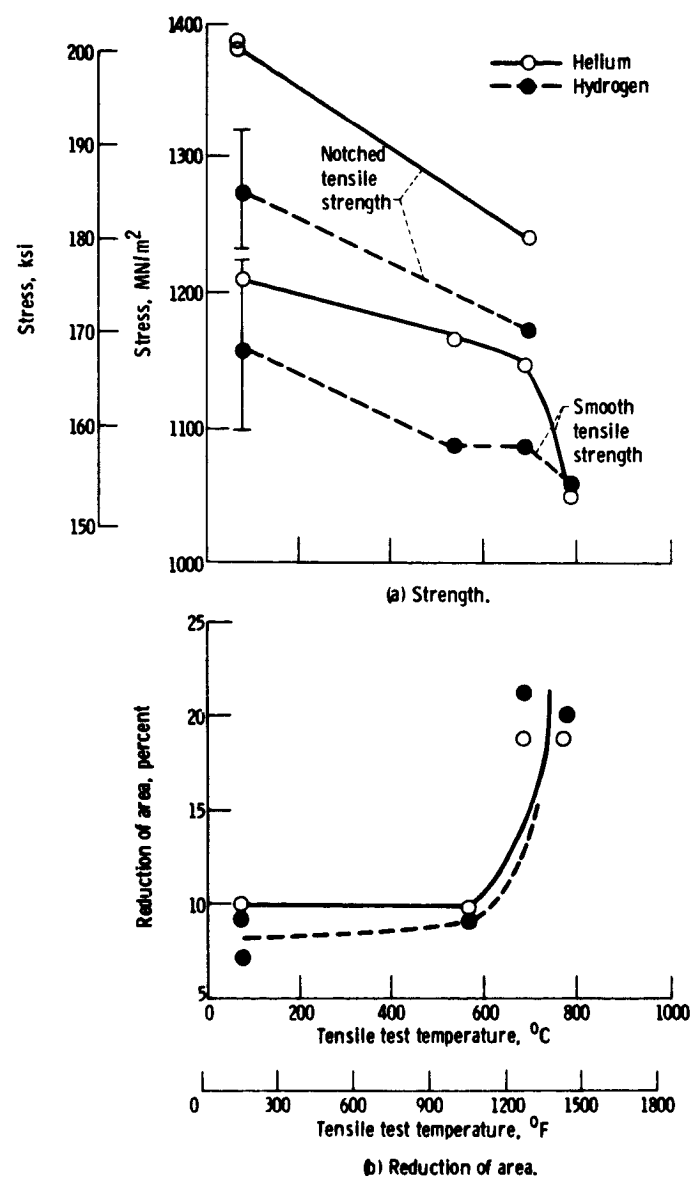
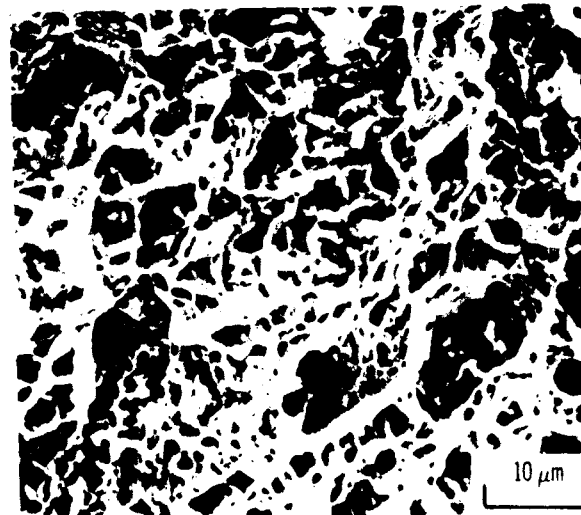
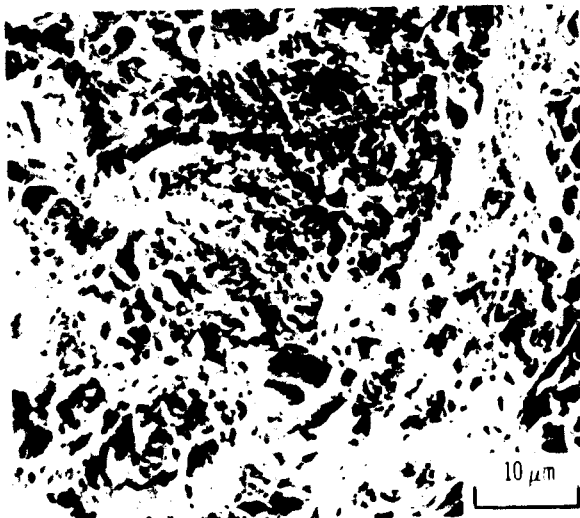
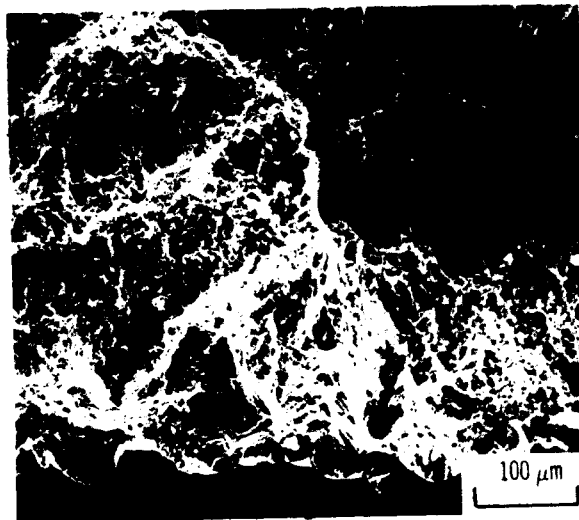
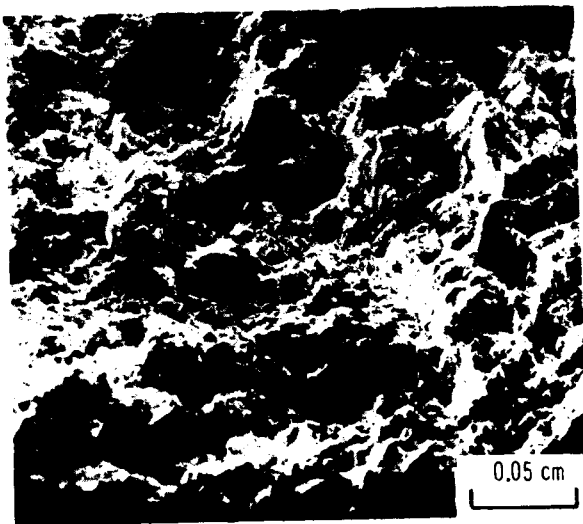


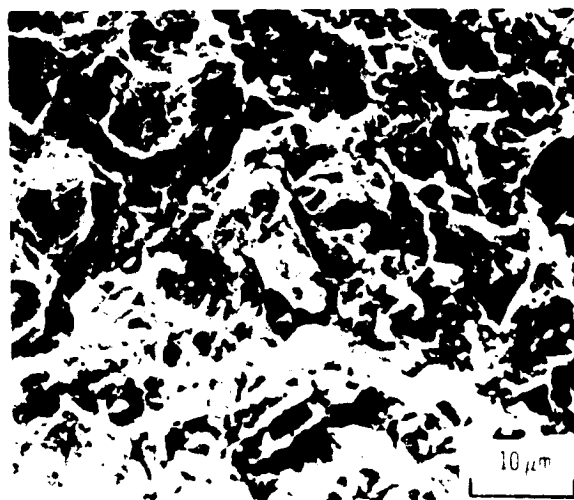
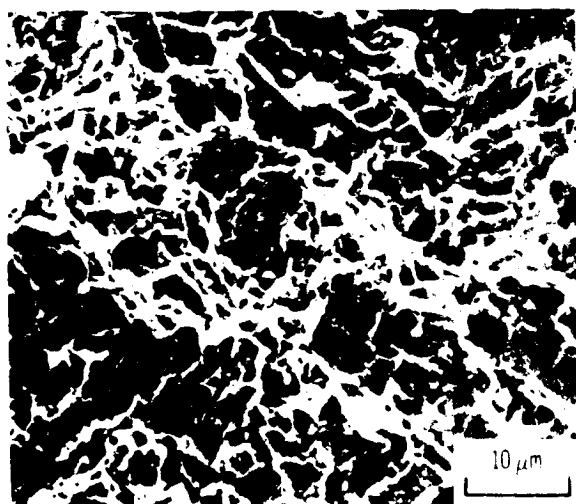
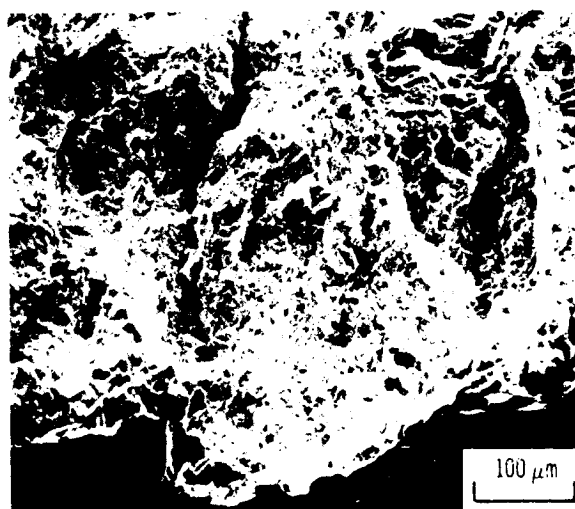
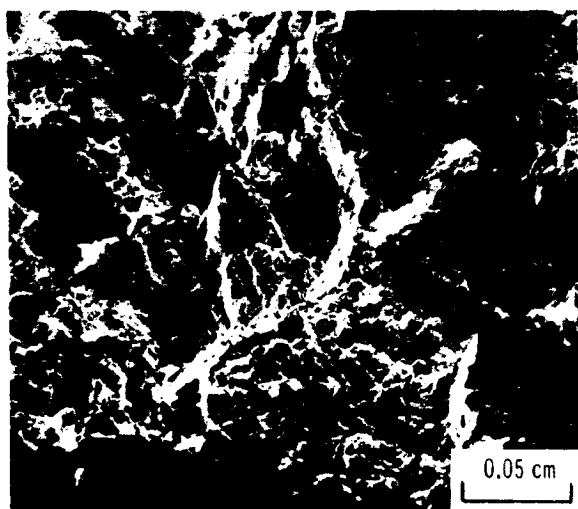
Figure 8. - Tensile properties of Astroloy in 3.5-MN/m² (500-psi) helium and hydrogen.



(a) Tested in helium; notched tensile strength, 1380 MN m^{-2} (200 ksi).

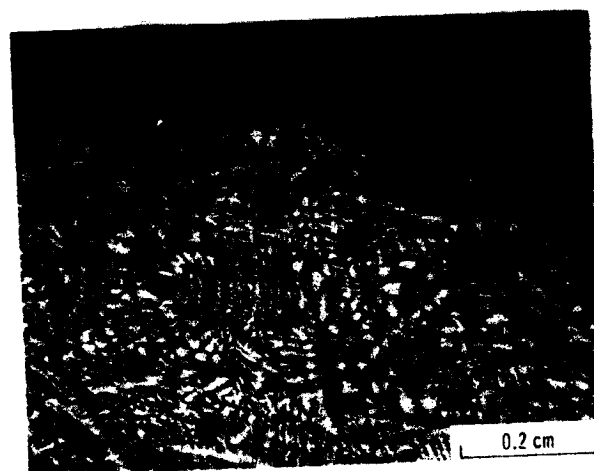
Figure 9. - Photomicrographs of Astroloy tested in 3.5-MN/m^2 (500-psi) helium or hydrogen at 23°C (73°F).

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(b) Tested in hydrogen; notched tensile strength: 1225 MN/m^2 (175 ksi).

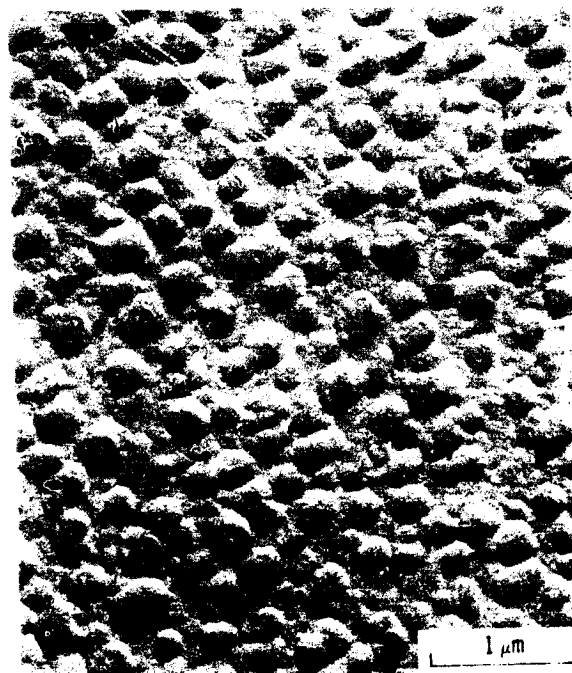
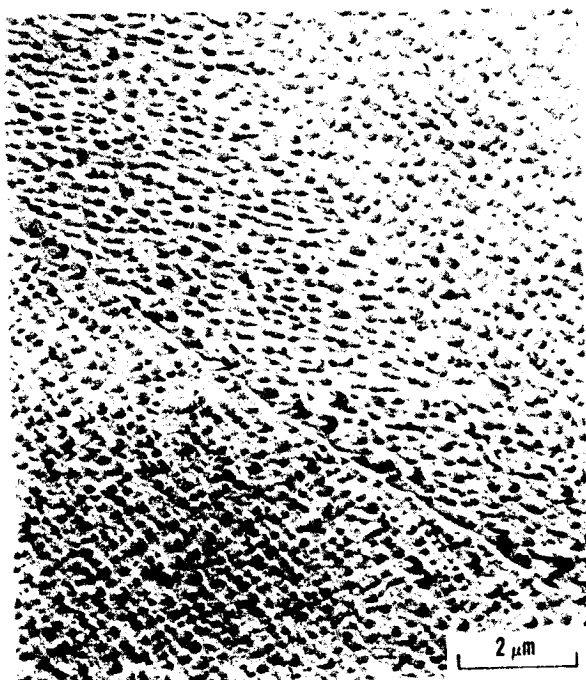
Figure 3 Continued



(c) Tested in helium; notched tensile strength, 1380 MN/m^2 (200 ksi).

(d) Tested in hydrogen; notched tensile strength, 1225 MN/m^2 (178 ksi).

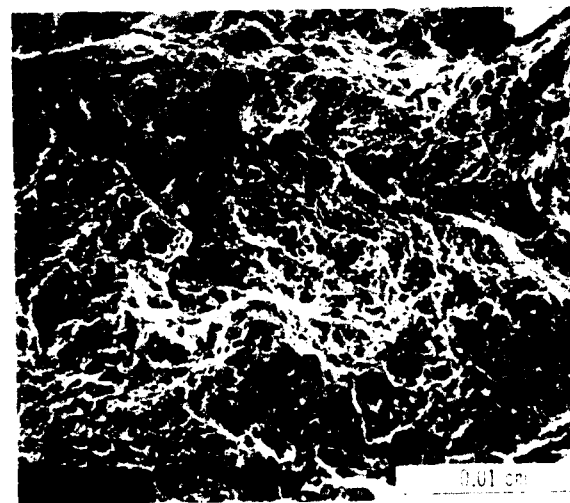
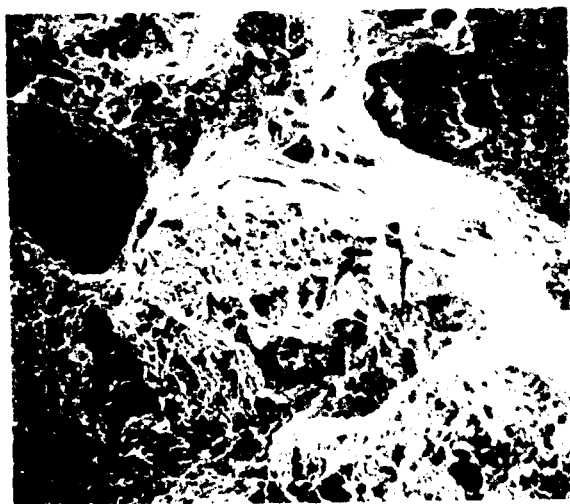
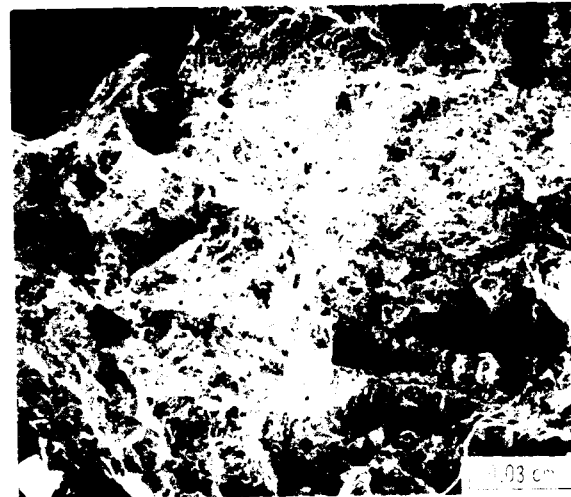
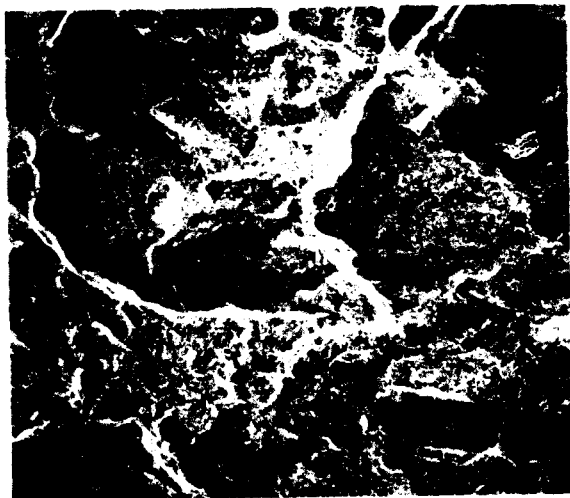
Figure 9. - Continued.



(e) Tested in hydrogen; notched tensile strength, 1225 MN m^2 (178 ksi).

Figure 9 - Concluded

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(a) Tested in helium; smooth tensile strength, 1215 MN/m² (176 ksi); 10-percent reduction of area.

(b) Tested in hydrogen; smooth tensile strength, 1095 MN/m² (159 ksi); 7-percent reduction of area.

Figure 10. - Scanning electron micrographs of Astroloy tested in 3.5-MN/m² (500-psi) helium and hydrogen at 23 °C (73 °F).

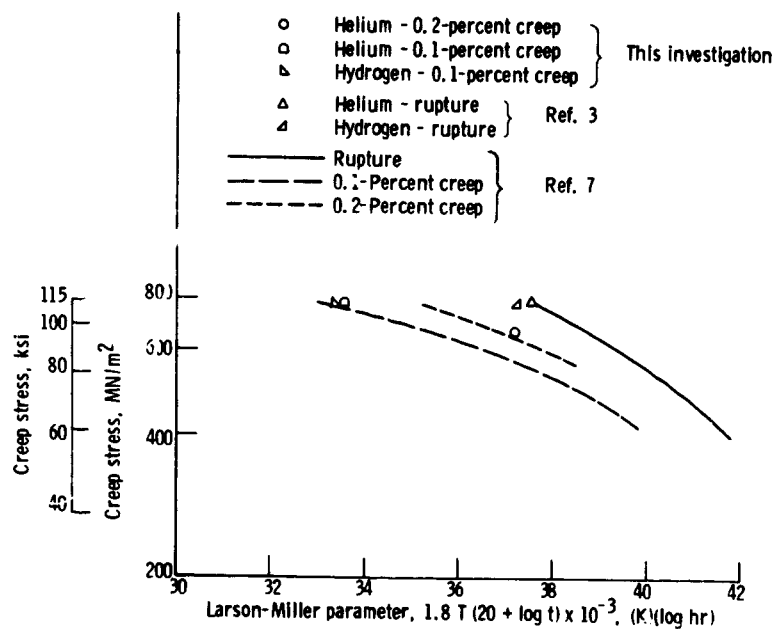


Figure 11. - Creep results for Astroloy in 3.5-MN/m² (500-psi) helium and hydrogen from this investigation and results in 35-MN/m² (5000-psi) hydrogen from reference 3.

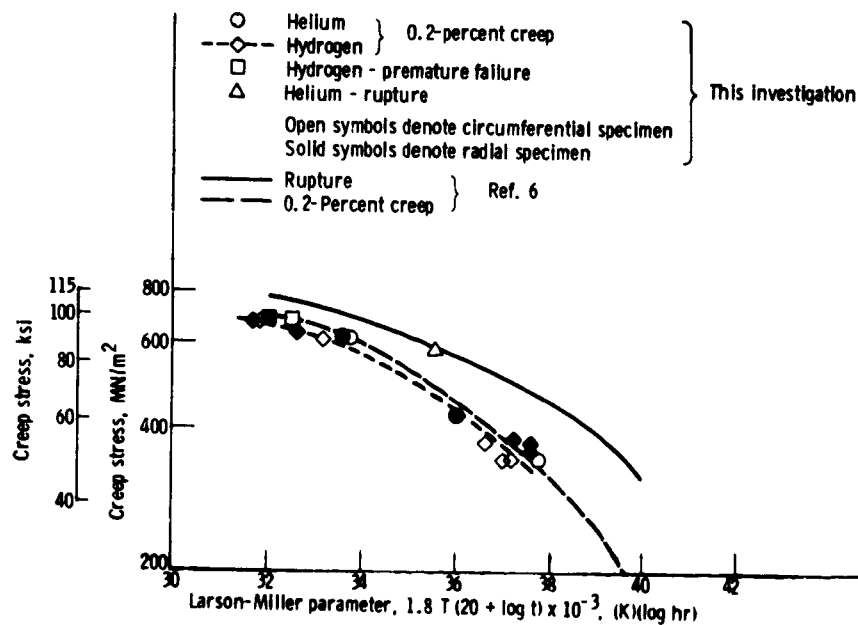


Figure 12. - Creep results for V-57 in 1.7-MN/m² (250-psi) helium and hydrogen.

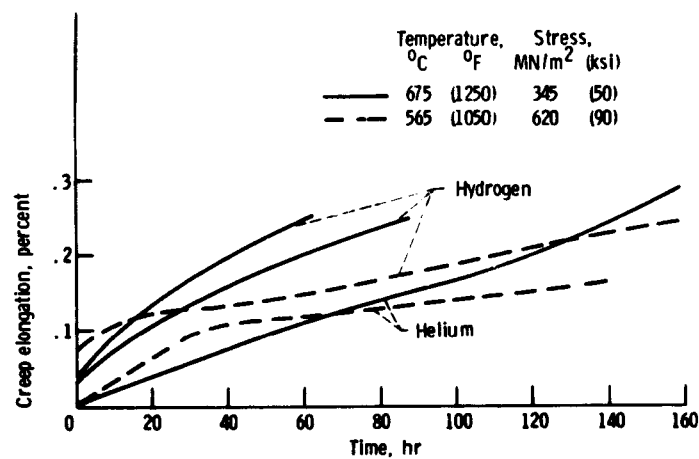


Figure 13. - Creep curves for V-57 in 1.7-MN/m² (250-psi) helium and hydrogen illustrating accelerated creep in hydrogen.

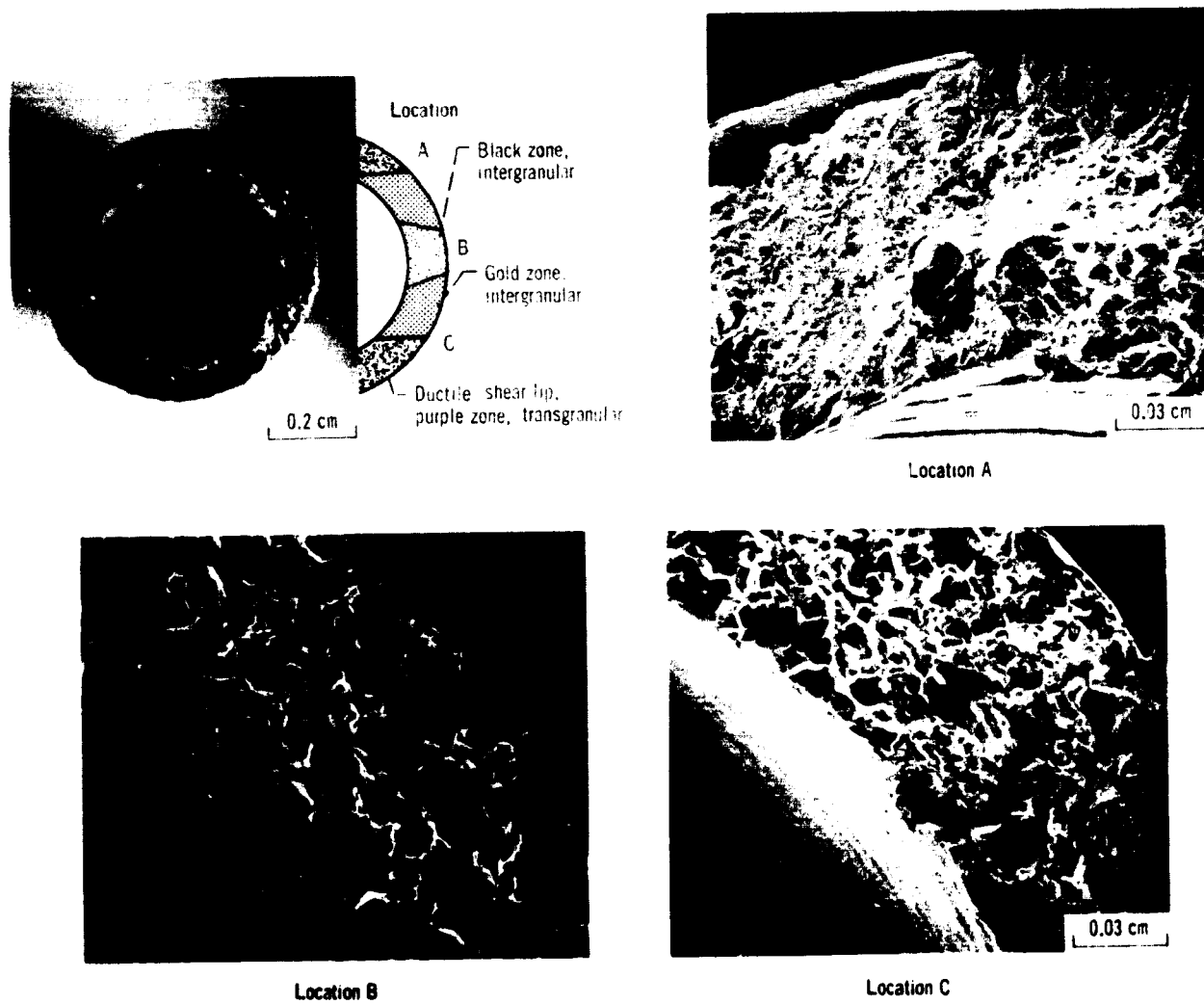
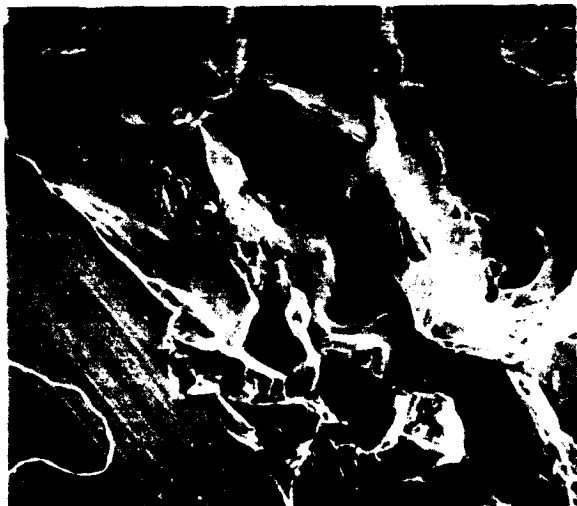
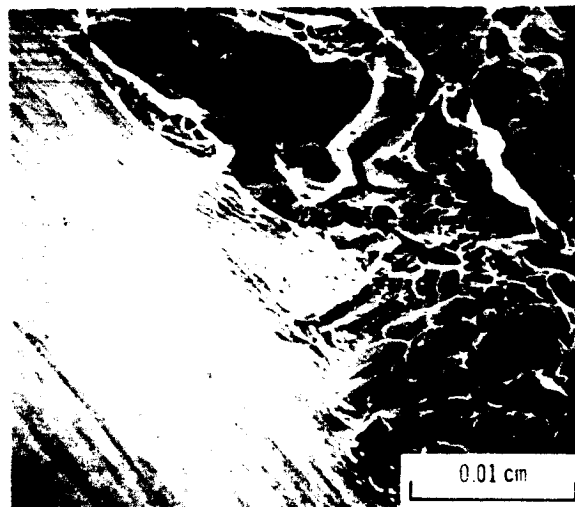


Figure 14. - Scanning electron micrographs of V-57 creep-rupture failure in hydrogen at 675 MN m^{-2} (98 ksi) and 565° C (1050° F).

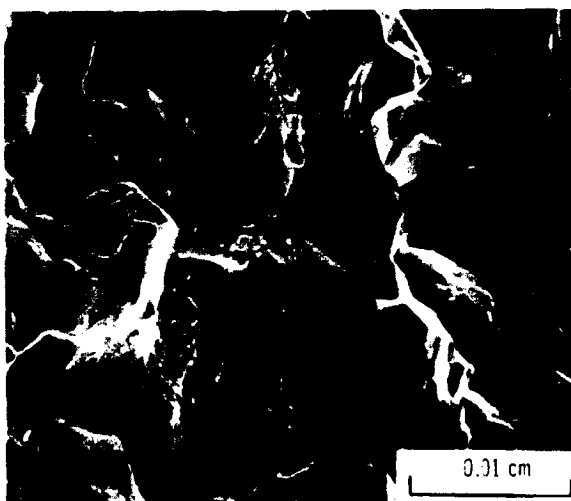
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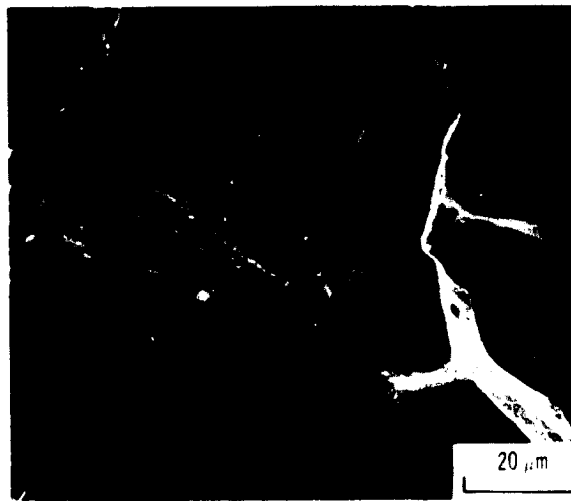
Location B



Location C



Location B



Location B

Figure 14. - Concluded.

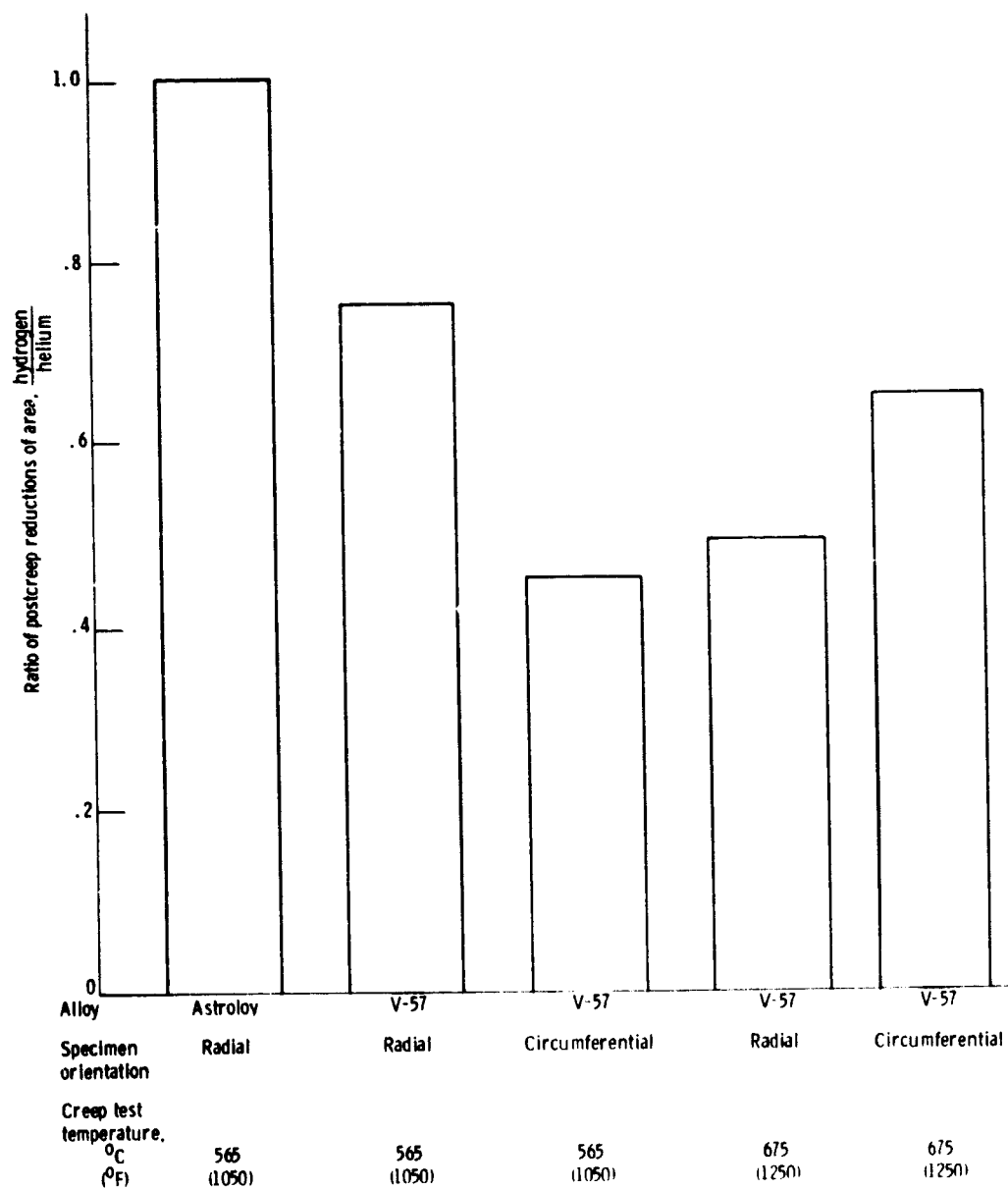
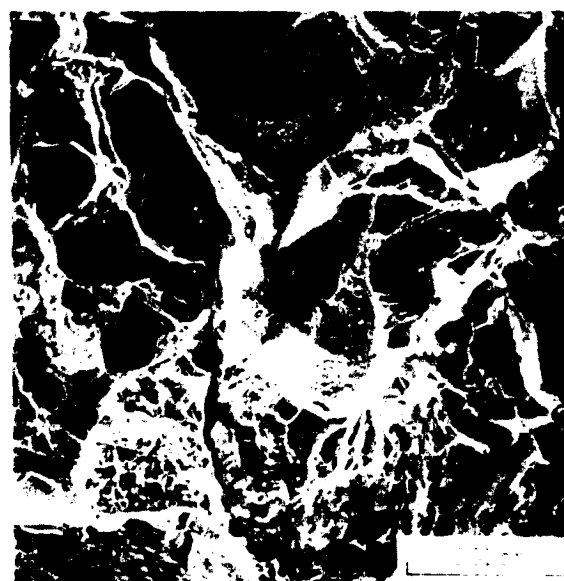
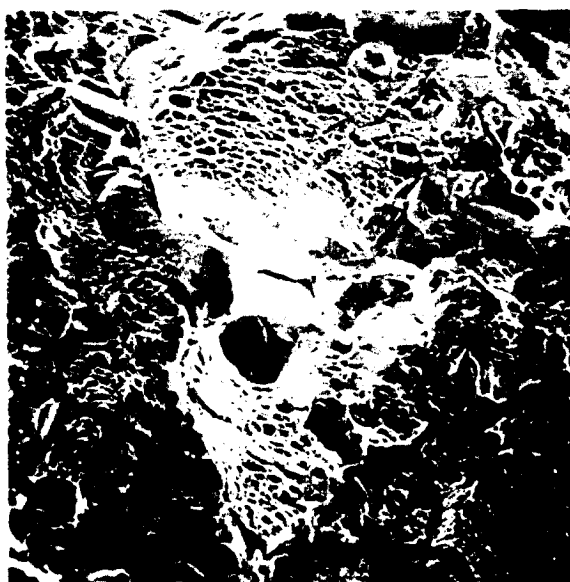
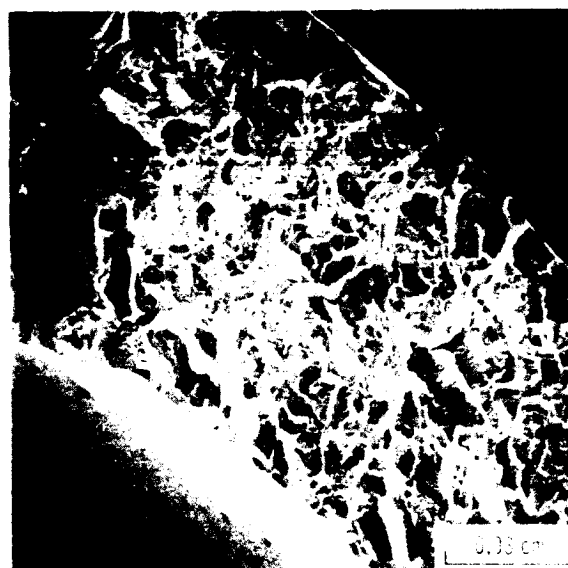
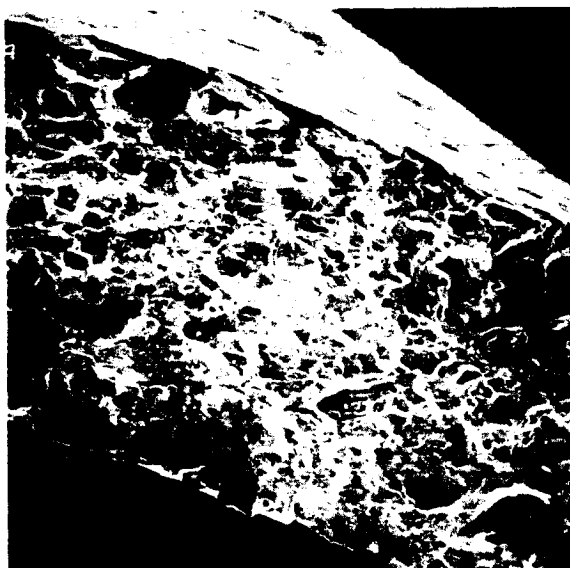


Figure 15. - Room-temperature reduction of area after prior creep exposure in hydrogen or helium.



(a) Creep in helium; 15-percent tensile elongation.

(b) Creep in hydrogen; 4-percent tensile elongation.

Figure 16. - Scanning electron micrographs of V-57 after creep and tensile tests at 23°C (73°F) in air.

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